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Explosives

Edward de Barry Barnett

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EXPLOSIVES

BY

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GENERAL PREFACE

THE rapid development of Applied Chemistry in recent years has brought about a revolution in all branches of technology. This growth has been accelerated during the war, and the British Empire has now an opportunity of increasing its industrial output by the application of this knowledge to the raw materials available in the different parts of the world. The subject in this series of handbooks will be treated from the chemical rather than the engineering standpoint. The industrial aspect will also be more prominent than that of the laboratory. Each volume will be complete in itself, and will give a general survey of the industry, showing how chemical principles have been applied and have affected manufacture. The influence of new inventions on the development of the industry will be shown, as also the effect of industrial requirements in stimulating invention. Historical notes will be a feature in dealing with the different branches of the subject, but they will be kept within moderate limits. Present tendencies and possible future developments will have attention, and some space will be devoted to a comparison of industrial methods and progress in the chief producing countries. There will be a general bibliography, and also a select bibliography to follow each section. Statistical information will only be introduced in so far as it serves to illustrate the line of argument.

Each book will be divided into sections instead of chapters, and the sections will deal with separate branches of the subject in the manner of a special article or monograph. An attempt will, in fact, be made to get away from

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the orthodox textbook manner, not only to make the treatment original, but also to appeal to the very large class of readers already possessing good textbooks, of which there are quite sufficient. The books should also be found useful by men of affairs having no special technical knowledge, but who may require from time to time to refer to technical matters in a book of moderate compass, with references to the large standard works for fuller details on special points if required.

To the advanced student the books should be especially valuable. His mind is often crammed with the hard facts and details of his subject which crowd out the power of realizing the industry as a whole. These books are intended to remedy such a state of affairs. While recapitulating the essential basic facts, they will aim at presenting the reality of the living industry. It has long been a drawback of our technical education that the college graduate, on commencing his industrial career, is positively handicapped by his academic knowledge because of his lack of information on current industrial conditions. A book giving a comprehensive survey of the industry can be of very material assistance to the student as an adjunct to his ordinary textbooks, and this is one of the chief objects of the present series. Those actually engaged in the industry who have specialized in rather narrow limits will probably find these books more readable than the larger textbooks when they wish to refresh their memories in regard to branches of the subject with which they are not immediately concerned.

The volume will also serve as a guide to the standard literature of the subject, and prove of value to the consultant, so that, having obtained a comprehensive view of the whole industry, he can go at once to the proper authorities for more elaborate information on special points, and thus save a couple of days spent in hunting through the libraries of scientific societies.

As far as this country is concerned, it is believed that the general scheme of this series of handbooks is unique, and it is confidently hoped that it will supply mental

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munitions for the coming industrial war. I have been fortunate in securing writers for the different volumes who are specially connected with the several departments of Industrial Chemistry, and trust that the whole series will contribute to the further development of applied chemistry throughout the Empire.

SAMUEL RIDEAL.

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PUBLISHERS' NOTE

We much regret that, owing to the continued and unexpected increase in the cost of printing since the Armistice, it has been found impossible to publish future volumes in the "Industrial Chemistry Series" at the price originally fixed of 7/6 each. If, as it is hoped, prices become more reasonable, we propose to revert as nearly as possible to the earlier arrangement.

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AUTHOR'S PREFACE

In the following pages the author has endeavoured to give a clear but concise account of the manufacture of explosives, together with an outline of the methods used for investigating this class of substance. The explosives industry is an important one, both in time of peace and in time of war, and is intimately bound up with the synthetic dyestuff and artificial fertilizer industries. These two latter industries are on the point of being established on what one trusts will be a secure basis in this country, and probably the explosives industry will expand with them. The experience gained by the troops and by the munition workers during the war in the handling and use of explosives should have largely removed the distrust in which these bodies are usually held, and at the same time have demonstrated their manifold uses. It is certain that prior to the war the use of explosives in this country was far too restricted, only small quantities being used for agricultural purposes, such as breaking up subsoil, drainage, etc. In the future it is hoped that they will be more extensively used for general purposes, and this will no doubt prove to be the case if they are made available at a low price. The enormous nitrating plants established for war purposes and the advent of synthetic ammonium nitrate should render this possible with nitrate of ammonia explosives. The author has devoted a special section to Coal Mine Explosives, as the importance of the subject seems to warrant special treatment. It is hoped that the British Government will divert some of the money that is to be expended on "Scientific Research" to the investigation of shot firing in coal mines,

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as up to the present they seem to have rested content with carrying out the official tests. It is rather depressing to find that, in spite of our enormous coal fields, we carry out less work on coal mine explosives than any of the important coal-getting countries in either hemisphere. There seems to be no British publication analogous to the excellent "Bulletins of the U.S. Bureau of Mines" or the "Bulletins of the U.S. Bureau of Explosives."

The author wishes to acknowledge his indebtedness to C. A. Marshall's invaluable work "Explosives; their History, Manufacture, Properties and Uses," in which the industry is treated in far greater detail than is possible in a volume of this size.

E. DE BARRY BARNETT.

June, 1919.

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ABBREVIATIONS

LITERATURE

A. *Annalen der Chemie.*
A.E. *Arms and Explosives.*
A.R. Annual Report of H.M. Inspector of Explosives.
B. *Berichte des Deutschen Chemischen Gesellschaft.*
C.r. *Comptes rendues.*
J.S.C.I. *Journal of the Society of Chemical Industry.*
P.S. *Memorial des Poudres et Salpêtres.*
Soc. *Transactions of the Chemical Society.*
S.R. Special Report by H.M. Inspector of Explosives.
S.S. *Zeitschrift f. Gesamte Schiess—u. Sprengstoff Wesen.*
D.R.P. Deutsches Reichs Patent.
A.P. American Patent.
E.P. English Patent.
F.P. French Patent.

COMPOUNDS, ETC.

The following abbreviations, which are current in all explosives works in Great Britain, are used throughout the text :—

B.G. Blasting Gelatine.
C.C. Collodion Cotton.
D.N.B. Dinitrobenzole.
D.N.N. Dinitronaphthalene.
D.N.T. Dinitrotoluol.
G.C. Guncotton.
G.P. Gunpowder.
M.J. Mineral Jelly.
M.N.B. Mononitrobenzole.
M.N.N. Mononitronaphthalene.
M.N.T. Mononitrotoluol.
N.C. Nitrocellulose.
N.G. Nitroglycerine.
P.A. Picric Acid.
T.N.A. Tetranitroaniline.
T.N.N. Trinitronaphthalene.
T.N.T. Trinitrotoluol.
W.M. Wood Meal.

M.A. is mixed acid (nitric and sulphuric) and N.A. nitric acid of any strength, the monohydrate being denoted by its chemical formula, HNO_3. S.A. is sulphuric acid of any strength, the commercial concentrated acid (168° Tw.) being denoted by C.O.V. (concentrated oil of vitriol), D.O.V. (double oil of vitriol) or R.O.V. (rectified oil of vitriol). The monohydrate is denoted by its chemical formula, H_2SO_4. The fuming acid is known as "oleum," or much less frequently as F.O.V. or N.O.V. (fuming oil of vitriol, Nordhausen oil of vitriol).

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EXPLOSIVES

INTRODUCTION

Historical Sketch.—The origin of gunpowder, which was the only explosive known until the middle of the nineteenth century, is uncertain. Some authors regard Greek Fire, rather extensively used in the defence of Constantinople in the seventh century, as a form of gunpowder, but it seems reasonably certain that this was merely an incendiary mixture to which crude nitre may or may not have been added in order to make it burn more fiercely. The Chinese seem to have made use of gunpowder in warfare about 1232, and the writings of Roger Bacon contain directions for the purification of nitre and anagrams and cryptograms which appear to describe the preparation of gunpowder, although some doubt has been cast on their authenticity. His later works, however, notably "Opus Tertium," "De Secretis," and "Opus Magnus," leave no doubt that he was acquainted with explosive mixtures of sulphur, charcoal, and nitre. About this period also, the Arabs seem to have had knowledge of the explosive properties of similar mixtures.

The early uses of gunpowder were confined to warfare, and no use seems to have been made of it for blasting purposes for several hundred years. It was originally used in the form of crude hand grenades, and probably was at first of most use in striking terror into the enemy. Cannon were first used by the English in 1346 at the battle of Crecy, but seem to have been used a few years prior to this in the Hispano-Moorish wars.

The manufacture of gunpowder was originally carried

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out by the very crude method of pounding the ingredients together by hand in mortars, but edge runners were introduced towards the end of the sixteenth century. The original mixtures were of very fine grain, were deliquescent, and the ingredients separated very easily when the powder suffered vibration as in transport. In the fourteenth century attempts were made to avoid this latter defect by the addition of camphor, sal-ammoniac and gum, and in the sixteenth century the process of "corning" or "granulating" was introduced. This was done by moistening the powder during the latter stages of mixing, so as to obtain a cake which was subsequently broken up and sifted.

Fuzed shell were first introduced in 1588, but the fuzes were naturally of a very crude nature. More accurate fuzes were employed by the British at the siege of Gibraltar in 1779, and shrapnel shell was introduced a few years later.

Berthollet, as a result of his researches on chlorates, in 1788 suggested substituting potassium chlorate for nitre, and obtained a more powerful explosive by this means, but it was too dangerous to make or use. A chlorate powder, however, was adopted in 1805 by Forsyth as a priming charge.

Fulminate caps seem to have come into use first about 1815, and in 1831 Bickford first introduced safety fuze. The discovery of guncotton by Schönbein in 1845, and of nitroglycerine by Sobrero in 1846, opened up new fields, although the accidents attendant on the manufacture of these substances at first greatly delayed their general introduction. Schönbein sold the British rights of his patent to John Hall and Sons, of Faversham, who manufactured guncotton for a few months, but abandoned it in 1847, after a disastrous explosion. Six years later the Austrian Government took up the matter, and General von Lenk constructed some batteries in which guncotton was used both as a propellant and as a bursting charge for the shell. These were not an unmitigated success, and after two disastrous explosions in 1865, they were abandoned.

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In the meantime the British Government had taken up the subject, and Frederick Abel carried out investigations on their behalf. He quickly realized that the instability of guncotton was due to the difficulty in washing it, and in 1865 introduced the pulping process. Simple as it may seem, this must be regarded as an epoch-making discovery, as it at once changed a very dangerous process into one of the safest known in an explosives works. Also the wet pulp could be compressed into blocks which were convenient for transport and use. The usefulness of these blocks was greatly enhanced by the discovery by Braun in 1868 that dry guncotton could be fired by a fulminate detonator, and that wet guncotton could be fired in the same way if a small primer of dry guncotton was used. The value of this discovery will be realized when it is remembered that wet guncotton is non-inflammable and insensitive to shock. These slabs of wet guncotton have for many years been the standard explosive for military demolitions and for signal maroons, and until quite recently have been the invariable charge for torpedoes.

As stated above, nitroglycerine was discovered in 1846 by Sobrero, who, however, does not seem to have attached any importance to the discovery, and it was not until Alfred Nobel took up the subject in 1862 that any attempt was made to employ it as an explosive. In this year Nobel commenced its manufacture near Stockholm in Sweden, but the numerous accidents that accompanied its use soon led to its prohibition by all countries. Nobel then sought for a means of rendering it more safe, and discovered in 1867 that this could be done by absorbing it in some porous material. The most suitable material of this nature was found to be kieselguhr, good qualities of which will take up three times their weight of nitroglycerine and still remain dry, although Nobel also patented the use of other porous materials, such as brick dust, plaster, etc.

A great advance was made in 1875, when Nobel introduced his first gelatinous explosive. The discovery of this is said to have been due to an accident, Nobel having used

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collodion solution to close a cut in his hand and then having noticed that this formed a jelly with the nitroglycerine with which he was carrying out some experiments later in the day. The first explosive of this nature, Blasting Gelatine, contained about 92 per cent. of nitroglycerine and 8 per cent. of collodion cotton. It experienced an immediate success, but a demand at once arose for less violent and less brisant explosives of the same nature. These were readily made by preparing a thinner jelly and then doping with potassium nitrate and wood meal. Mixtures of this nature have met with very wide use under the names of Gelatine Dynamite and Gelignite, Gelatine Dynamite being stronger than Gelignite, but not so strong as Blasting Gelatine.

A new class of explosive was introduced by Sprengel in 1871. He found that mixtures of suitable organic matter, such as nitrobenzole, and strong or fuming nitric acid or liquid nitrogen tetroxide, could be detonated. These had the advantage that the ingredients were kept separate and only mixed just before the shot was fired, and although nitric acid is an inconvenient liquid to transport, Sprengel explosives enjoyed some vogue. A more rational explosive of the Sprengel class was introduced by Devine in 1880. This consisted in a cartridge of potassium chlorate which immediately before use was dipped into an organic liquid such as nitrobenzole. Rock-a-rock was an explosive of this nature, and was used in 1885 for blasting Hell Gate Rock in New York harbour. The first ammonium nitrate explosive was introduced by Favier in 1885, but attracted scant attention at the time on account of its objectionable hygroscopic properties. Finally, in recent years proposals have been made to employ mixtures of liquid air or oxygen in conjunction with organic matter, and although a good deal of the blasting in connection with the Simplon tunnel was carried out with explosives of this class, they suffer from obvious disadvantages and are never likely to come into general use. They have been used, however, for general blasting purposes in Germany during the war.

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It may be mentioned that explosives of the Sprengel class have never been used in this country, as mixing the ingredients is regarded as a process of manufacture within the meaning of the Act, and as such can only be carried on on licensed premises (Explosives Act, 1875).

As stated on page 2, Berthollet in 1788 was the first to propose the use of chlorates, but was compelled to abandon the scheme on account of the great sensitiveness of his mixtures. This danger was remedied by Street in 1897, who prepared safe chlorate mixtures from potassium chlorate and castor oil thickened with a nitrohydrocarbon. These have met with considerable success under the name of Cheddite.

The first smokeless powder after von Lenk's nitrocotton batteries was introduced by Schultze in 1865. It consisted of pellets of wood which were nitrated and then impregnated with barium nitrate and/or potassium nitrate. A great stride was made by Volkmann five years later, who partly gelatinized nitrated wood by treatment with a mixture of alcohol and ether. This must be regarded as having laid the foundation of the modern smokeless powder industry. A similar powder, E.C. Powder, was introduced in 1882 by the Explosives Company of Stowmarket. It consisted of partly gelatinized nitrocotton impregnated with a mixture of barium and potassium nitrates, and found immediate favour among sportsmen.

The first smokeless military powder was Poudre B, introduced by Vielle in 1886, and adopted by the French Government. It was made by forming a dough of nitrocellulose with alcohol and ether, which was then rolled out into sheets, cut up into strips, and dried.

Two years later Nobel introduced Ballistite, made by gelatinizing soluble nitrocellulose with nitroglycerine, and in the same year the British Government introduced Cordite, made by gelatinizing guncotton with nitroglycerine and acetone. These rival discoveries led to a lawsuit, the issue of which turned on the definition of nitrocellulose.

Sprengel in 1871 drew attention to the fact that picric acid could be detonated, but no use was made of this

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discovery until Turpin proposed its use in 1886 as a bursting charge for shells. For this purpose it found wide application under the names of Melinite, Lyddite, etc., but has now been largely replaced by the cheaper and safer trinitrotoluol.

Tetranitroaniline and tetranitromethyl aniline were discovered by Flürsheim in 1910, but at present their cost of manufacture precludes their use except in detonators, in which they are likely to find extensive employment.

The general introduction of explosives for blasting purposes led to a considerable increase in coal-mine accidents, due to the shot firing the mine gases or causing a dust explosion. Macnab in 1873 first suggested remedying this by placing a cylinder of water in front of the charge, and this was shortly followed by suggestions involving the employment of jellies containing 90 per cent. of water, wet moss or salts rich in water of crystallization in the same way. None of these were altogether successful, but the setting up of experimental galleries in 1885 by the Prussian and other governments soon led to the discovery that as a rule ammonium nitrate explosives are the safest to use in fiery mines. The conditions which must be fulfilled by an explosive for use in fiery mines are now stringent, and in order to fulfil them it is necessary to add a considerable amount of an inactive salt to the explosive. It was not until 1913 that a gelatinous explosive was discovered which would pass the Rotherham test, in which year Messrs. Curtis and Harvey, Ltd., patented Super-Rippite.

Explosives Act, 1875.—The Explosives Act, 1875, is entitled "An Act to amend the law with respect to manufacturing, keeping, selling, carrying and importing gunpowder, nitroglycerine and other explosive substances." The third section of the Act defines an explosive as ". . . gunpowder, nitroglycerine, blasting powders, fulminate of mercury or of other metals, coloured fires, and every other substance, whether similar to those above mentioned or not, used or manufactured with a view to produce a practical effect by explosion or a pyrotechnic effect; and includes fog-signals, fireworks, fuzes, rockets, percussion caps, detonators,

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cartridges, ammunition of all descriptions, and every adoption or preparation of an explosive as so defined." Section 104 of the Act gives power to include any specially dangerous substance even if not manufactured or used "to produce a practical effect by explosion." Acetylene, for example, under certain conditions is included under the Act. The Explosives Act prohibits the manufacture of any explosive on any premises not duly licensed, fixes the minimum distance of such premises from private dwellings, public roads, etc., and establishes procedure for granting such licences. It provides safeguards for workers, insists that each separate building in a factory shall be separately licensed and the amount of explosive and the number of workers in it at any time limited. It prohibits certain dangerous processes and mixtures, such as mixtures of chlorates and sulphur, and endows the inspectors under the Act with wide powers. The Act must be regarded as a very fair one, and is administered with extremely little friction in view of the stringency of its provisos.

Buildings and Safety.—Owing to the dangerous nature of the material handled, manufacturing operations are carried out in a series of small separate huts, the number of workers in any hut rarely exceeding five, although in some of the less dangerous processes this number may be exceeded. The danger buildings in an explosives factory can be roughly divided into two classes, viz. (*a*) magazines used for storing explosives, and (*b*) buildings used for manufacturing purposes. The former of these should be of strong construction, so as to prevent the entrance of any unauthorized person. They are usually built of stone or brick, with double doors, the outer door generally being sheathed in iron. Working buildings, on the other hand, should be of as light and flimsy a character as possible, so that in case of an explosion heavy debris is not thrown about. They are usually constructed of light matchboard, the roof being of the same material and covered with some non-inflammable sheet. A light, ferroconcrete construction has been proposed as suitable for danger buildings, as it has been urged that an

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explosion would reduce it to powder, but the suggestion has not been adopted.

All danger buildings must be surrounded by an earth mound reaching to the roof, so as to deflect the explosive wave upwards in case of an explosion. The entrance through the mound should be directly opposite the door of the building when possible. The floors of danger buildings are usually covered with oilcloth, or in some cases with sheet lead. They must be washed thoroughly every day, and when dusty material such as dry guncotton is being dealt with must be kept wet while work is being carried on. All windows must be made of frosted glass so as to exclude direct sunlight, and all doors must open outwards. Further, the doors must be provided with a brass lock and key, for securing them when work is not being carried on, but when occupied by workers must be closed only by a spring catch, so that they can be opened by a push in case of emergency. The brass work must either be kept bright or painted over. Artificial light when required is provided by incandescent electric lamps. These are enclosed in a glass bell and then set in recesses in the walls, and separated from the inside of the building by a thick plate of glass. All switches, etc., must be external to the building. All workers must wear special clothes made of wool and without pockets, and must carry with them no metallic articles, such as metal buttons, jewellery, etc., nor any smoking materials or matches. Danger buildings are all "clean" buildings, and must not be entered unless the person entering dons special shoes. Workers usually wear slippers without nails, felt slippers or rubber boots. These are kept in a cupboard in the porch of the building, and are donned just before stepping over the barrier, so that they never come into contact with the ground outside the clean building. The barrier is usually a strip of wood about eight inches high, set at the door of the building to denote the point at which the clean area commences. Magazines and some other buildings can be entered by wearing leather overshoes kept specially for the purpose, but this is not permitted in all buildings. As some

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explosives are easily electrified, it is advisable that workers' shoes should be provided with a few copper studs in the sole, so as to "earth" the wearer. This is particularly the case when reeling cordite and when handling dry gun-cotton. Of course all buildings must be adequately protected against lightning, and when possible work must be stopped during thunderstorms.

The manufacture of explosives as carried out in this country must be regarded as one of the safest of the dangerous trades, the average number of fatalities per annum for the decade 1904–1914 being 7·7. The following is a summary of the number of accidents, deaths, and injuries which occurred in manufacturing operations in the explosives trade during the years 1911–1914 inclusive :—

Year.	No. of Accidents.	Killed.	Injured.
1911	69	13	40
1912	104	1	33
1913	86	13	50
1914	92	21	41

In relation to the number of accidents, it must be borne in mind that any accident due to explosion, no matter how trivial, must be reported. During the war the number of accidents and the number of killed and injured greatly exceeded these figures, but this must be ascribed to the enormously increased production, accompanied by intensive working and dilution of labour.

Imports and Consumption.—The consumption of explosives in Great Britain is chiefly supplied by the various explosive factories in the country, but some explosive is imported. The following figures denote the chief blasting explosives imported in 1912 and 1913, but a large proportion was only imported in transit and was not consumed :—

	1912.	1913.
Blasting Gelatine	17,512 lbs.	240,393 lbs.
Dynamite	116,370 ,,	218,741 ,,
Gelignite	280,488 ,,	686,995 ,,

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No data is available as regards the consumption of sporting powder, but the consumption of blasting explosives is reported under the Mines and Quarries Act, and the figures for 1910 and 1911 give a good idea of the extent of the industry—

	1910.	1911.
Gunpowder	17,664,483 lbs.	17,305,073 lbs.
Permitted Explosives ..	8,607,882 ,,	9,340,033 ,,
Gelignite	3,039,256 ,,	3,294,423 ,,
Gelatine Dynamite ..	494,560 ,,	472,464 ,,
Blasting Gelatine ..	257,756 ,,	244,155 ,,
Cheddite	123,584 ,,	110,761 ,,
Other Explosives ..	350,600 ,,	530,979 ,,
Total	30,538,121 ,,	31,297,888 ,,

In the United States the industry is much larger than in this country, the home consumption for the years 1914–1917 being—

	1914.	1915.	1916.	1917.
Black Blasting Powder	206,099,700	197,722,300	215,575,025	277,118,525
High Explosives ..	218,453,971	235,828,587	255,154,787	262,316,080
Permissible Explosives	25,697,818	27,349,909	34,685,240	43,040,722
Total	450,251,489	460,900,796	505,415,052	582,475,327

all amounts being in pounds. Large as these figures are, they do not include the very considerable export trade carried on, the figures for which during the corresponding years were—

Year.	Dynamite, etc.		Gunpowder.		Cartridges.	Other explosives.
	Weight (lbs.)	Value (U.S. Dollars.)	Weight (lbs.)	Value (U.S. Dollars.)	Value (U.S. Dollars.)	Value (U.S. Dollars.)
1914	11,296,115	1,213,600	896,569	291,453	6,567,122	1,965,412
1915	11,446,368	1,509,050	84,358,379	66,922,807	25,408,079	95,129,957
1916	18,601,285	4,173,175	303,648,981	262,116,893	55,103,904	394,136,334
1917	17,930,665	3,653,374	446,540,999	331,163,229	42,122,656	255,944,315

Classification.—The following is the British Official classification of explosives extracted verbatim from the "List of Authorized Explosives," 1918:—

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Class I.—Gunpowder

The term "gunpowder" means exclusively gunpowder ordinarily so called.

Class II.—Nitrate Mixture

The term "nitrate mixture" means any preparation, other than gunpowder ordinarily so called, formed by the mechanical mixture of a nitrate with any form of carbon or with any carbonaceous substance not possessed of explosive properties, whether sulphur be or be not added to such preparation, and whether such preparation be or be not mechanically mixed with any other non-explosive substance.

Every blasting explosive of this class, in which nitrate of ammonium, nitrate of sodium or chloride of sodium are used as ingredients, shall be contained in cartridge wrappers or cases (or in 5 lb. inner packages) made thoroughly waterproof with melted paraffin or other suitable waterproofing material.

Class III.—Nitro-Compound

The term "nitro-compound" means any chemical compound possessed of explosive properties, or capable of combining with metals to form an explosive compound, which is produced by the chemical action of nitric acid (whether mixed or not with sulphuric acid) or of a nitrate mixed with sulphuric acid upon any carbonaceous substance, whether such compound is mechanically mixed with other substances or not.

The nitro-compound class has two divisions.

Every explosive of this class and every explosive ingredient thereof shall be so thoroughly purified and otherwise of such a character as to satisfy a test known as the Heat Test, and specified in a Memorandum signed by a Government Inspector and dated the 2nd of February, 1914.*

Every blasting explosive in this class, in which nitrate of ammonium, nitrate of sodium or chloride of sodium are

* See Section IX.

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used as ingredients, shall be contained in cartridge wrappers or cases (or in 5 lb. inner packages) made thoroughly waterproof with melted paraffin or other suitable waterproofing material.

Division 1 comprises any chemical compound or mechanically mixed preparation which consists either wholly or partly of nitroglycerine or of some other liquid nitro-compound.

Provided that every explosive in this Division shall be of such a character and consistency as not to be liable to liquefaction or exudation.

Provided also that an explosive that is required by definition to be issued in waterproof inner packages may be exempted from such requirement by Special Authority, when and so long as the conditions of such Authority are observed.

Division 2 comprises any nitro-compound as before defined, which is not comprised in the first division.

Class IV.—Chlorate Mixture

The term "chlorate mixture" means any explosive containing a chlorate.

The chlorate mixture class has two divisions.

Every explosive of this class, and every explosive ingredient thereof, shall be so thoroughly purified and otherwise of such a character as to satisfy a test known as the Heat Test, and specified in a Memorandum signed by a Government Inspector and dated the 2nd February, 1914.*

Every blasting explosive in this class in which nitrate of ammonium, nitrate of sodium or chloride of sodium are used shall be contained in cartridge wrappers or cases (or in 5 lb. inner packages) made thoroughly waterproof with melted paraffin or other suitable waterproofing material.

Division 1 comprises any chlorate preparation which consists partly of nitroglycerine or of some other liquid nitro-compound.†

Provided that every explosive in this Division shall be

* See Section IX.

† No explosive of this division is at present authorized.

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of such a character and consistency as not to be liable to liquefaction or exudation.

Division 2 comprises any chlorate mixture as before defined, which is not comprised in the first division.

Class V.—Fulminate

The term "fulminate" means any chemical compound or mechanical mixture, whether included in the foregoing classes or not, which, from its great susceptibility to detonation, is suitable for employment in percussion caps or any other appliances for developing detonation, or which, from its extreme sensibility to explosion, and from its great instability (that is to say, readiness to undergo decomposition from very slight exciting causes), is especially dangerous.

This class consists of two divisions.

Division 1 comprises such compounds as the fulminates of silver and of mercury, and preparations of these substances, such as are used in percussion caps; and any preparation consisting of a mixture of a chlorate with phosphorus, or certain descriptions of phosphorus compounds, with or without the addition of carbonaceous matter, and any preparation consisting of a mixture of a chlorate with sulphur, or with a sulphuret, with or without carbonaceous matter.*

Division 2 comprises any such substance as the chloride and the iodide of nitrogen, fulminating gold and silver, diazobenzol, and the nitrate of diazobenzol.†

Class VI.—Ammunition

The term "ammunition" means an explosive of any of the foregoing classes when enclosed in any case or contrivance, or otherwise, adapted or prepared to form a cartridge or charge for small arms, cannon, or any other weapon, or for blasting, or to form any safety or other fuze for blasting, or for shells, or to form any tube for firing

* The only explosive of this division at present authorized is mercury fulminate.

† The only explosive of this division at present authorized is lead azide.

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explosives, or to form a percussion cap, a detonator, a fog-signal, a shell, a torpedo, a war rocket, or other contrivance other than a firework.

The term "percussion cap" does not include a detonator.*

The term "detonator" means a capsule or case which is of such strength and construction and contains an explosive of the fulminate explosive class in such quantity that the explosion of one capsule or case will communicate the explosion to other like capsules or cases.

The term "safety fuze" means a fuze which burns and does not explode, and which does not contain its own means of ignition, and which is of such strength and construction and contains an explosive in such quantity that the burning of such fuze will not communicate laterally with other like fuzes.

The ammunition class has three divisions.

Division 1.—No official definition of Division 1 is given, but it includes safety electric fuzes, percussion caps and railway fog-signals.

Division 2 comprises any ammunition as before defined which does not contain its own means of ignition, and is not included in Division 1.

Division 3 comprises any ammunition as before defined which contains its own means of ignition and is not included in Division 1.

Class VII.—Firework

The term "firework" comprises firework composition and manufactured fireworks.

Division 1.—Firework composition.†

Division 2.—Manufactured fireworks, consisting of any

* A percussion cap can only be properly classed as such if it contains less than ·6 grain of a composition of the 1st Division of the 5th (Fulminate) class of which not more than 25 per cent. consists of fulminate of mercury, or less than ·5 grain of any other explosive of the 1st Division of the 5th (Fulminate) class. And it has been further decided that percussion caps shall not be classed as such when they contain anvils or have their composition unprotected by tinfoil or other suitable substance.

† No explosive of this division is at present authorized.

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explosive of the foregoing classes, and any firework composition, when such explosive or composition is enclosed in any case or contrivance, or is otherwise manufactured so as to form a squib, cracker, serpent, rocket (other than a war rocket), maroon, lance, wheel, and Chinese fire, Roman candle, or other article specially adapted for the production of pyrotechnic effects or pyrotechinc signals, or sound signals. Provided that a substantially constructed and hermetically closed metal case containing not more than 1 lb. of coloured fire composition of such a nature as not to be liable to spontaneous ignition shall be deemed to be a manufactured firework. Provided also that the term "manufactured firework" shall not be deemed to include any explosive hereinafter mentioned, by whatever name known, or any colourable imitation of the same.

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A bibliography of the chief works on ballistics will be found at the end of the section on Propellants.

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Very valuable information is also published from time to time in the "Bulletins" and "Technical Papers" of U.S. Bureau of Mines and the U.S. Bureau of Explosives.

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Section I.—GUNPOWDER

Gunpowder remained the only known explosive until the middle of the nineteenth century, and at the present time is still the most used explosive except for military purposes. For military purposes its use is now limited to bursting charges for shrapnel shell, to time fuzes, and to priming composition for use with cordite, but it is still largely used as a sporting powder, chiefly on account of its cheapness. The same consideration, and the fact that it does not require a detonator, has led to its retention for blasting purposes, about 55 per cent. of the explosive used for blasting in Great Britain in 1911 being gunpowder.

Compared with modern high explosives Gunpowder is greatly lacking in power, the Trauzl block test giving figures of about 108 as compared with 520 for Dynamite No. 1 and 650 for Blasting Gelatine. Gunpowder has the advantage, however, of being less brisant than most explosives, a great advantage when dealing with soft material which it is desired to obtain in big lumps.

Gunpowder invariably consists in this country of a mixture of potassium nitrate, sulphur, and charcoal, although in America and in Germany large quantities are consumed in which the cheaper sodium nitrate has been substituted for the potassium salt. These sodium nitrate powders are cheaper and somewhat more powerful than those made with potassium nitrate, but their deliquescent properties are a great drawback. They have been used in Great Britain since the outbreak of war cut off the potash supplies from Germany.

In manufacturing gunpowder some care is required in the selection of material. The nitre should be almost chemically pure, be quite neutral to litmus, and quite free

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from chlorate and perchlorate, especially the former. It should not contain more than the merest trace of chloride, under ·01 per cent. calculated as NaCl, as otherwise it will become deliquescent.

The sulphur must contain no non-volatile matter, and must be free from sulphuric acid. As commercial flowers of sulphur almost invariably contains some acid, it is usual to use roll sulphur and grind this down. The quality of the powder depends a great deal on the quality of the charcoal, so that it is customary for powder mills to prepare their own. The wood most employed in this country is alder wood and dog wood, but hazel, willow, yew, hemp, and poplar are all used. The German cocoa powder, one of the best gunpowder propellants for cannon ever made, was prepared from rye straw charcoal. It has, however, been completely superseded by the modern smokeless powders.

Timber intended for gunpowder charcoal should be cut in the spring, and then kept for from three months to three years in order to allow the sap to evaporate. The bark is then removed and the wood cut up and carbonized in iron cylinders.

Various forms of retorts have been proposed, but the most generally used is in the form of a cylinder 4 ft. 6 in. long by 2 ft. 4 in. diameter. These are charged with the wood, and then closed, except for a vent to allow the escape of the products of distillation. The retort is then slid into the furnace and carbonization carried out for 4 to 5 hours at a temperature between 400° C. and 500° C. The crude pyroligneous acid that distils off can be collected and worked up for acetic acid, acetone, etc., but the amounts are so small that the distillate is usually led direct to the furnace and burnt. When carbonization is complete the retort is removed from the furnace, completely closed and set aside to cool. Air must not be admitted until the charge has cooled, and even then only slowly, as freshly made charcoal absorbs air with avidity, and may inflame. It is for this reason that movable retorts are usually preferred to fixed ones, although both types are used.

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The *composition* of gunpowder varies a good deal with the different grades manufactured. French military powders remained of the same composition from 1598 until the adoption of the modern smokeless powders. They were made according to the famous recipe "As, as, six," viz. 1 part of sulphur, 1 part of charcoal, and 6 parts of nitre. This corresponds to nitre 75 per cent., and sulphur and charcoal 12·5 per cent. each. The following table gives the composition of a few modern powders made by different countries, but it must be borne in mind that various grades of powder are in use :—

Country.	Sporting powder.			Blasting powder.		
	KNO_3	S	C	KNO_3	S	C
Great Britain	75	10	15	65–75	10–20	15
France { Ordinaire ..	—	—	—	62	20	18
France { Lente ..	78	10	12	40	30	30
France { Forte ..	—	—	—	72	13	15
Germany	78	10	12	65–70	14–20	16
Austria	76	9·4	14·6	60·2	18·4	21·4

In the manufacture of gunpowder the ingredients are first ground separately in any suitable form of mill. Charcoal should not be ground for at least a fortnight after burning, as otherwise it may inflame through the absorption of oxygen. Sulphur in grinding requires care, as it is easily electrified, and consequently sulphur mills should be carefully earthed. Precautions should also be taken against a dust explosion, and the same applies to charcoal.

The actual manufacture of gunpowder in a modern factory involves seven operations, or eight in the case of moulded powders. The object in the rather elaborate procedure is to obtain a uniform powder, which will not separate and the grains of which will not crumble.

First Mixing.—This is carried out either in a copper ball mill with lignum vitæ balls, or in a revolving drum through which passes a shaft carrying eight arms or "flyers." The drum and shaft revolve in opposite directions, the former making 60 revolutions per minute and the latter 120. A uniform mixture is obtained in a few minutes, and the "green" charge then emptied out, sifted, and removed to the incorporating house.

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Incorporating or Milling.—This was formerly carried out in stamp mills, and these are still in use in some of the smaller continental works, but are being rapidly replaced by edge runners. These consist of stone runners acting on a stone bed, or of iron runners on an iron or hard wood bed. Stone must not run on iron, or *vice versâ*, on account of the danger of sparks. The runners weigh 4 to 5 tons each, and are set at different distances from the driving shaft, so that they do not run on the same path. Each is provided with a scraper to prevent its picking up cake, and with a phosphor bronze or hard wood plough to push the charge into its path. In the best type of incorporating mill the runners do not rest on the bed, but are suspended a short distance above it, and each is capable of an independent vertical movement, so as to allow it to pass over any extra hard lumps without undue friction (Figs. 1 and 2). In Germany this arrangement is compulsory if iron runners and an iron bed are used. A charge for incorporation is 50–80 lbs. and requires from 3–8 hours, depending on the nature of the powder. Moisture, in the form of condensed steam from the drying stoves, is added from time to time so as to maintain a

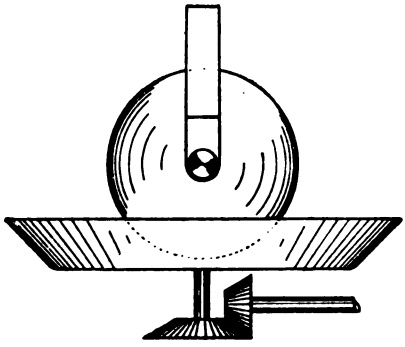

FIG. 1.—Gunpowder Incorporating Machine

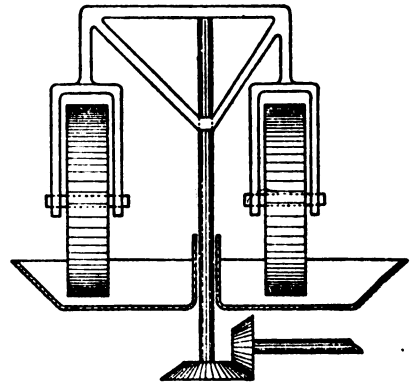

FIG. 2.—Gunpowder Incorporating Machine.

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water content of 3–6 per cent. After incorporation is complete, the charge is removed and roughly broken up by hand, and is then ready for pressing. Any hard incrustation that remains in the incorporating mill must be thoroughly soaked in water, and then removed with a wooden tool. Incorporation is a somewhat dangerous process, and in this country special safety arrangements must be provided. These consist of a water tank over each machine connected with a large board directly above the incorporator, so that any movement of the board caused by an explosion wave tips the whole contents of the tank on to the charge and drowns it. All the tanks in each house are interconnected, so that the movement of any board actuates them all. Usually six machines are placed in each house, each machine being separated by a strong partition of masonry. Should the charge in any one mill explode, the force of the explosion wave acting on the board above it tips all the tanks in the building, and so prevents the explosion spreading.

Pressing.—This is carried out in hydraulic presses capable of dealing with about 1000 lbs. at a time. The presses are charged as follows. A copper or ebonite plate is laid in a horizontal position and surrounded by a light wooden frame about 1 in. deeper than the thickness of the plate. The tray thus formed is filled with the powder to be pressed, and this carefully smoothed off level with the top of the wooden frame. Another plate is then placed on the top of this, and this surrounded by another wooden frame, and the tray thus formed filled with powder. This is repeated until the full number of plates have been used, usually 20–30. The size of the plates varies in different factories, but they are usually about 2 ft. 4 in. by 1 ft. 8 in. The wooden frames are then removed and the pile slid into an hydraulic press with upward moving ram (Fig. 3). The pressure applied is about 400 lbs. per square inch, but it is best measured by watching the movement of the ram and not by gauges. It must be applied slowly, the time occupied being about 2 hours. When pressing is complete the plates

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are picked off one by one, and the outer edge of each cake to a depth of about 1½ in. cut off and kept separate from the main bulk. This is done as the periphery has not sustained the full pressure. The cuttings are given a short milling and then repressed, whereas the main bulk passes on to the granulating machine.

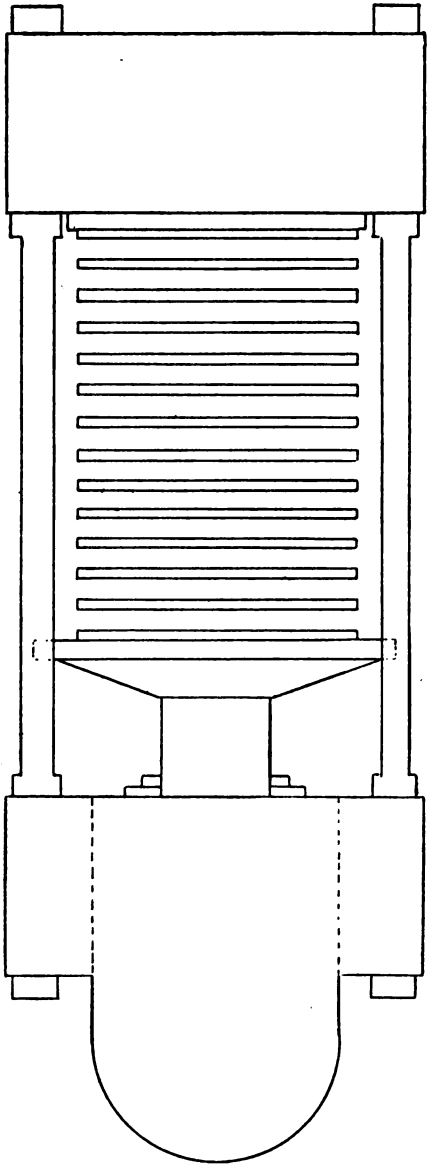

FIG. 3.—Gunpowder Press.

Pressing is a somewhat dangerous process, and an explosion is usually very disastrous on account of the large quantity of powder being dealt with. In order to protect the workers all controls should be in a special room separated from the press by a strong wall, arrangements being made for the workman to observe the movement of the ram without entering the press room.

In this country plates of ebonite are preferred to those of copper, as they are not so easily deformed and transmit the pressure more evenly. On the other hand they are easily electrified, and Guttmann records

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one fatal explosion in which the workman observed a spark 4 in. long pass from his hand to the press. Needless to say, presses should be carefully earthed.

Granulating or Corning.—In this process the press

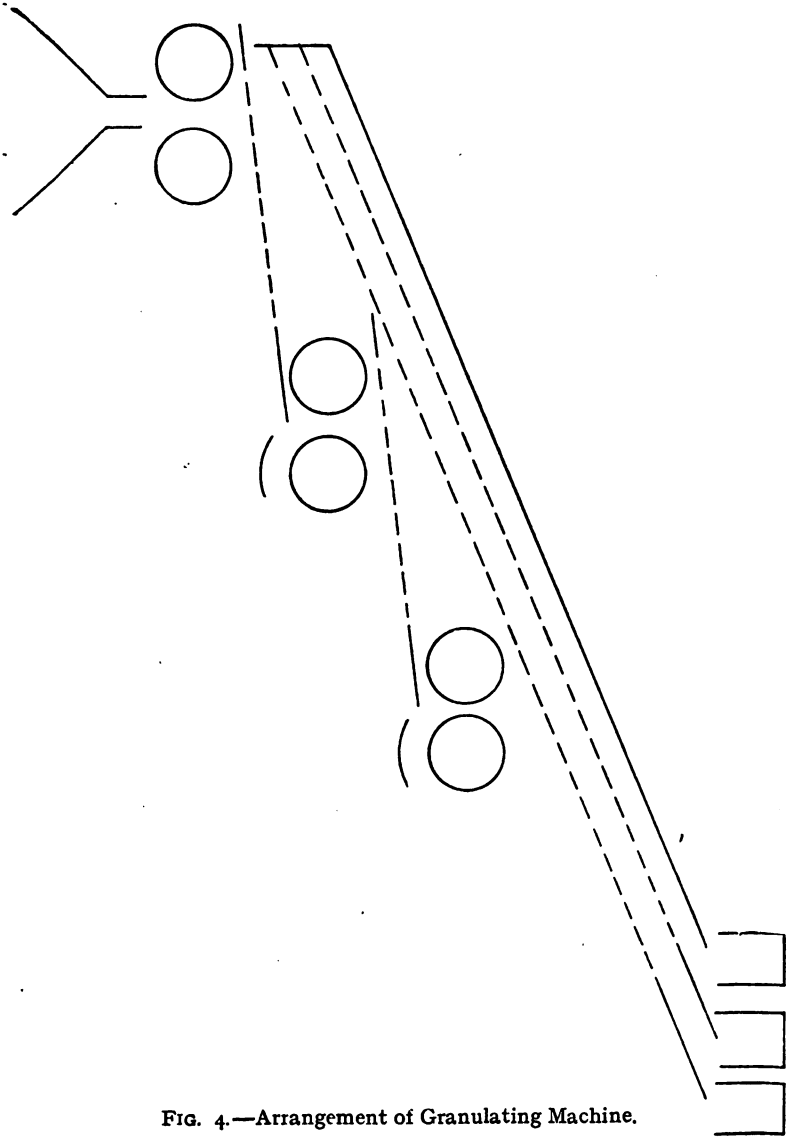

FIG. 4.—Arrangement of Granulating Machine.

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cakes are broken up into grains of the desired size by passing them through a series of three or more pairs of toothed gun-metal rollers revolving towards one another. Between each pair of rollers are vibrating screens of gun-metal wire which separate the powder into three sizes, viz. oversize, which passes on to the next set of rollers; right size, which is collected for the next stage; and undersize, which is collected and remilled and pressed. The construction of a granulating machine is shown diagrammatically in Fig. 4. The rollers should be mounted with spring bearings, so that if they encounter an extra hard piece of cake they can open and let it pass (Fig. 5). Powders made from dogwood

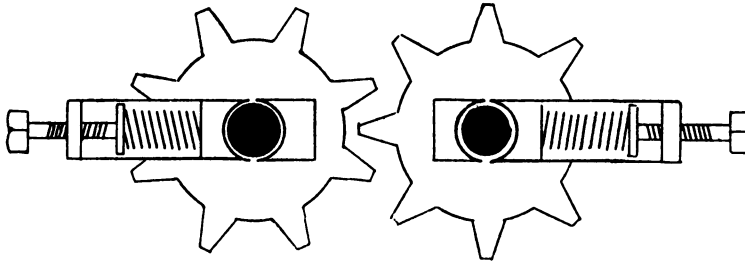

FIG. 5.—Rollers of Granulating Machine. Detail showing Spring Bearings.

charcoal usually produce a great deal of dust, and are generally given an extra screening ("dusting") to remove this.

Glazing.—This is carried out in revolving wooden drums, and has the effect of rounding off the grains and improving their appearance. A little graphite is sometimes added when slow-burning powders are being made.

Stoving or Drying.—This is carried out by spreading the powder in thin layers on trays, consisting of a wooden frame with a canvas bottom. Drying is carried out at 40° C. by blowing air heated by means of hot water pipes into the building.

Blending.—This consists in mixing several batches so as to obtain a uniform product.

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Moulding.—Before smokeless powders were introduced suitable slow-burning powders for use with rifled ordnance were prepared by compressing the blended gunpowder into cubes, pyramids, or prisms with from one to eight perforations. The German cocoa powder was especially suited for making moulded powders, as it flowed well in the die. The perforations had the object of obtaining a more constant rate of combustion. These moulded powders are no longer manufactured, but moulded cartridges are used for blasting purposes. These are simply prepared by measuring a definite amount of powder into a die and then compressing it by means of two pistons, one working upwards and one downwards, actuated by mechanical or hydraulic means. The lower piston carries a plunger which enters a prepared hole in the upper piston, and so leaves a perforation in the cartridge in which the fuze can be inserted.

Sodium Nitrate Powders.—Powders in which the potassium nitrate has been replaced by the cheaper sodium salt are used in gigantic quantities in America. They are somewhat more powerful than ordinary gunpowder, but their chief attraction lies in their cheapness. They suffer from the great disadvantage, however, of being very hygroscopic. They are made in much the same way as ordinary powder, and have an average composition—

Sodium nitrate	74
Sulphur	10
Carbon	16

Although the annual consumption of this type of powder in the United States amounts to nearly a hundred million pounds (50,000 short tons), it was not used in Great Britain until the outbreak of war in 1914 cut off the supply of potash and compelled the Government to alter the definition of gunpowder, so as to include powders made with sodium nitrate. A very similar powder is much used in Germany under the name "*Sprengsalpeter.*" It is composed of—

Sodium nitrate	75
Brown coal (lignite)	15
Sulphur	10

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It is regarded as safer than ordinary powder, and in Germany can be sent in unrestricted amount by goods train.

"*Bobbinite*" is a form of gunpowder, and is the favourite coal-getter in this country. It does not pass the Rotherham test, but its use under certain restrictions was specially permitted for a term of five years from January 1st, 1914, and by a further Order in Council dated September 18th, 1918, this period has been extended until December 31st, 1920. It is defined in the Explosives in Coal Mines Order of September 1st, 1913, as having the composition—

Ingredients.	1st definition.		2nd definition.	
	Maximum.	Minimum.	Maximum.	Minimum.
Potassium Nitrate ..	65	62	66	63
Charcoal	19·5	17	20·5	18·5
Sulphur	2·5	1·5	2·5	1·5
Ammonium Sulphate ..	17	13	—	—
Copper Sulphate				
Rice or Maize Starch ..	—	—	9	7
Paraffin Wax	—	—	3·5	2·5
Moisture	2·5	—	3	—

It is manufactured in moulded pellets, each pellet being coated with paraffin wax and wrapped in brown paper. Bobbinite made according to the first definition must have a density not exceeding 1·42, and according to the second definition not exceeding 1·48. There is no charge limit, and it must *not* be fired with a detonator.

Stability, etc.—Gunpowder is to be regarded as a very safe explosive. It ignites when heated at 270°–300° C., and is fired when struck by a two-kilogram weight falling a distance of 70–100 cm., the powder being confined between hardened steel surfaces. It is completely ruined by water, and powder that has been damp does not regain its full strength on drying. This is due to the moisture dissolving part of the nitrate which crystallizes out on drying, and spoils the intimate mixing of the ingredients so necessary in order to obtain a good explosive. On explosion, it gives about 44 per cent. of gaseous and 56 per cent. of solid products. The gaseous products of explosion are chiefly

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nitrogen, carbon dioxide and carbon monoxide, together with small quantities of hydrogen, methane, and sulphuretted hydrogen. The solid products are chiefly composed of carbonate and sulphate of potassium, together with some unburnt sulphur and traces of sulphide, thiocyanate, and nitrate of potassium.

One gram of powder yields from 250 to 300 cc. of permanent gas measured at N.T.P., and evolves from 500 to 700 calories. The temperature of explosion is probably about 2700° C.

LITERATURE

The best account of the history of gunpowder is given by Col. Hime in "Gunpowder and Ammunition." London, 1904.

A full description of the manufacture of charcoal and sulphur will be found in R. Escales, "Schwartzpulver u. Sprengsalpeter," Leipzig, 1914; and in O. Guttmann, "Manufacture of Explosives," London, 1895.

Information on the products of explosion will be found in the following papers:—

A., **102**, 325; **109**, 53; **265**, 257.

Trans. Roy. Soc., 1875, 49.

Numerous accidents have happened during the manufacture of gunpowder, but the following Special Reports are selected as being particularly instructive: S.R., 160, 165, 177, 179, 181, 189, 190, 202.

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Section II.—EXPLOSIVE COMPOUNDS

The number of explosive compounds suitable for industrial use is very limited, owing to the difficulty in combining suitable physical properties with sufficient stability and cheapness of manufacture. Up to the present, with the exception of fulminate of mercury and azide of lead, both of which are largely used for detonators and percussion caps, the only explosive compounds that have been found to fulfil industrial requirements are esters of nitric acid and aromatic nitro-compounds. Of the former the esters of the lower alcohols are too volatile for use, while those of the higher alcohols are too expensive and are deficient in oxygen. Of the polyhydric alcohols glycerine trinitrate is far the most used, as glycerine can be obtained at a moderate price and its trinitrate is easily prepared and is insoluble in water, so that it can be readily washed free from acid. For special purposes the tetranitrate of diglycerine and the dinitrate of monochlorhydrin are used. In France the use of glycol dinitrate has been proposed, as it has been stated that glycol can be obtained from acetylene at a price which enables it to compete with glycerine. Tartaric acid forms a dinitrate, but unfortunately it is too unstable for use as an explosive.

The other polyhydric alcohols are too expensive to allow of their nitrates finding industrial application, although the use of mannitol hexanitrate has been proposed. If these polyhydric alcohols could be obtained at a reasonable price their nitrates would form valuable explosives, as the percentage of available oxygen increases with the number of carbon atoms carrying a hydroxyl group. Thus, glycol dinitrate contains no available oxygen—

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$$\begin{matrix}CH_2ONO_2\\ |\\ CH_2ONO_2\end{matrix} = CO_2+N_2+2H_2O$$

whereas glycerine trinitrate contains 3·5 per cent. and mannitol hexanitrate contains 7·1 per cent.—

$$2CH_2ONO_2.CHONO_2.CH_2ONO_2=6CO_2+5H_2O+3N_2+O$$
$$CH_2ONO_2.(CHONO_2)_4.CH_2ONO_2=6CO_2+4H_2O+3N_2+O_2$$

The sugars form nitric esters, and attempts have been made to use glucose nitrate and sucrose nitrate. Unfortunately, the nitric esters of the sugars are soluble in water and are hygroscopic and very sensitive. Polyhydric cyclohexanols such as inosite form nitric esters, inosite forming a hexanitrate, but unfortunately inosite and its derivatives are merely chemical curiosities at present.

Of the fatty hydroxyl compounds of unknown molecular weight cellulose and starch are the best known, and both form nitric esters. Of these the cellulose nitrates are very widely used (guncotton, pyroxylin, collodion cotton), and are usually made from cotton, although wood cellulose is used to some extent. There are mechanical difficulties in nitrating starch and stabilizing the product which have prevented the use of starch nitrates up to the present time.

Of the nitro-compounds those of the aliphatic series would be useful if they could be obtained more readily, tetranitromethane, for example, containing nearly 24 per cent. of available oxygen—

$$C(NO_2)_4 = CO_2+2N_2+3O_2$$

Unfortunately they are all troublesome and expensive to make, and those having hydrogen atoms attached to the same carbon atom as the nitro-group have the objectionable property of forming unstable salts with metals—

$$CH_3NO_2 \rightarrow \begin{matrix}H\\ \diagdown\\ \quad C=N\\ \diagup\\ H\end{matrix}\begin{matrix}O\\ \diagup\!\!\diagup\\ \diagdown\\ OM\end{matrix}$$

A large number of aliphatic nitro-compounds also have objectionable toxic properties, especially tetranitromethane.

Of the aromatic hydrocarbons benzene forms a dinitro-

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compound easily, and a trinitro-compound with such great difficulty that it is little more than a chemical curiosity. Toluol, on the other hand, forms a trinitro-compound with ease, and the same is the case with xylol. Toluol is also fairly easily nitrated in the side chain (E.P. 6076[11]), and readily yields nitrophenyl nitromethane. The tetranitro-compound, trinitrophenyl nitromethane, does not, however, seem to have been studied. Of the other aromatic hydrocarbons naphthalene gives a tetranitro-compound, but only with some difficulty, so that the mono-, di- and tri-nitro-compounds only are used.

The entrance of nitro-groups into the nucleus is facilitated by the presence of amino and hydroxyl groups. Thus, aniline gives a tetranitro-compound with some difficulty, and methyl aniline gives a tetranitro-compound (trinitrophenylmethyl nitramine), which although somewhat expensive for ordinary purposes, are used for detonators. Diphenylamine gives a hexanitro-compound which has found some application, but unfortunately the nitro-groups confer strongly acidic properties on the imino hydrogen atom.

Phenol forms a trinitro-compound (picric acid), and so does cresol. Both have been used, but both are, unfortunately, strongly acidic.

No attempts seem to have been made to utilize compounds such as nitrobenzyl nitrate, although these should be readily obtained by nitrating benzyl alcohol. Nitric esters are always less stable than nitro-compounds, so that substances containing both groups might be useful for detonators if for no other purpose.

The aromatic diazo salts are all explosive, but as a rule are too sensitive and too deficient in oxygen to be of any practical value. Attempts have been made, however, to use *p*-nitrodiazobenzene nitrate for detonators, but without much success.

It should be noted that all aromatic nitro-compounds are very deficient in oxygen. Even the unknown hexanitro-benzene would only contain just sufficient oxygen for its complete combustion. Hence, although some of the

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nitro-compounds can be detonated alone, they are almost invariably used in conjunction with an oxidizing agent such as ammonium nitrate, although for certain military purposes picric acid and trinitrotoluol are used unmixed with other substances.

The organic peroxides are also explosives, but are far too unstable to be of any practical value, and the same applies to the ozonides. These latter are rich in oxygen; benzene ozonide, for example, has the formula $C_6H_6O_9$, and would be useful were it not for their extreme instability.

Of the inorganic explosive compounds, lead azide is used to some extent for detonators as a substitute for mercury fulminate. The chlorates being formed endothermically from the chloride and oxygen are mildly explosive, and are used in some explosive mixtures, *e.g.* Cheddite, and in fireworks. Ammonium nitrate can be detonated with great difficulty, and is used to a very large extent. Ammonium perchlorate can also be detonated, and is an ingredient of some explosives. Ammonium bichromate is also an explosive, and although somewhat expensive and dangerous, is used in one or two French powders. The capacity of all these ammonium salts to explode is due to the acid radical oxidizing the ammonium radical.

NITROGLYCERINE

The term "nitroglycerine" is chemically incorrect, the explosive having the formula $CH_2(ONO_2)CH(ONO_2)CH_2(ONO_2)$, and being therefore glycerine trinitrate. The term "nitroglycerine" is, however, invariably used commercially, and in practice is frequently abbreviated to N.G.

Glycerine for nitration should be almost pure. Good dynamite glycerine has only a pale amber colour, and its specific gravity should be not less than 1260. It should be almost free from ash and contain only slight traces of fatty acids, aldehydes (acrolein), unsaponified fat, chlorides, sulphates, etc. It should contain no sugar or glucose, although these are sometimes present as adulterants. For the nitration only dearsenicated or non-arsenical acids should be used.

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Nitration is invariably carried out with a mixture of nitric and sulphuric acid, and nowadays it is customary to make up the mixed acids with oleum, so that the mixture is almost anhydrous, but contains no free sulphuric anhydride. The use of oleum is not essential, but is a matter of convenience. The composition of the mixed acid varies in different factories, but the following are all in use with satisfactory results :—

HNO_3	40	46·5	41·5
H_2SO_4	58·4	52·5	58·0
H_2O	1·6	1·4	·5

About 6 parts of mixed acid are used to every part of glycerine.

In some countries nitration is carried out in cast-iron jacketed vessels provided with a mechanical agitator. These are prohibited in almost all European countries, lead vessels with cooling coils being used and agitation being effected by compressed air. In Great Britain nitration must be carried on at a temperature not exceeding 22° C., but in other countries the limit is 25° C., or even 30° C. The temperature is observed by means of two thermometers, one dipping into the charge and one suspended above the charge and registering the temperature of the "gas," *i.e.* the air which is being forced through the charge for agitating it.

Nitrating House.—Two types of nitrator are in general use, but the older type introduced by Nobel (Fig. 6) is being rapidly displaced by the nitrator-separator of Thomson, Nathan, and Rintoul. In the older type of plant the charge of acid is run in from the measuring tank, the air agitation and cooling water turned on and the glycerine then sprayed in, the rate at which the glycerine is added being regulated so as to maintain a temperature of 22° C. During the nitration the workman watches the fumes through the window A, any appearance of red fumes being a sign of danger. During the nitration the run-off cock, B, is temporarily connected by means of a movable lead gutter with a large lead-lined drowning tank outside the building, so that

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should the charge get out of control it can be at once drowned. The provision of a drowning tank is only a precautionary

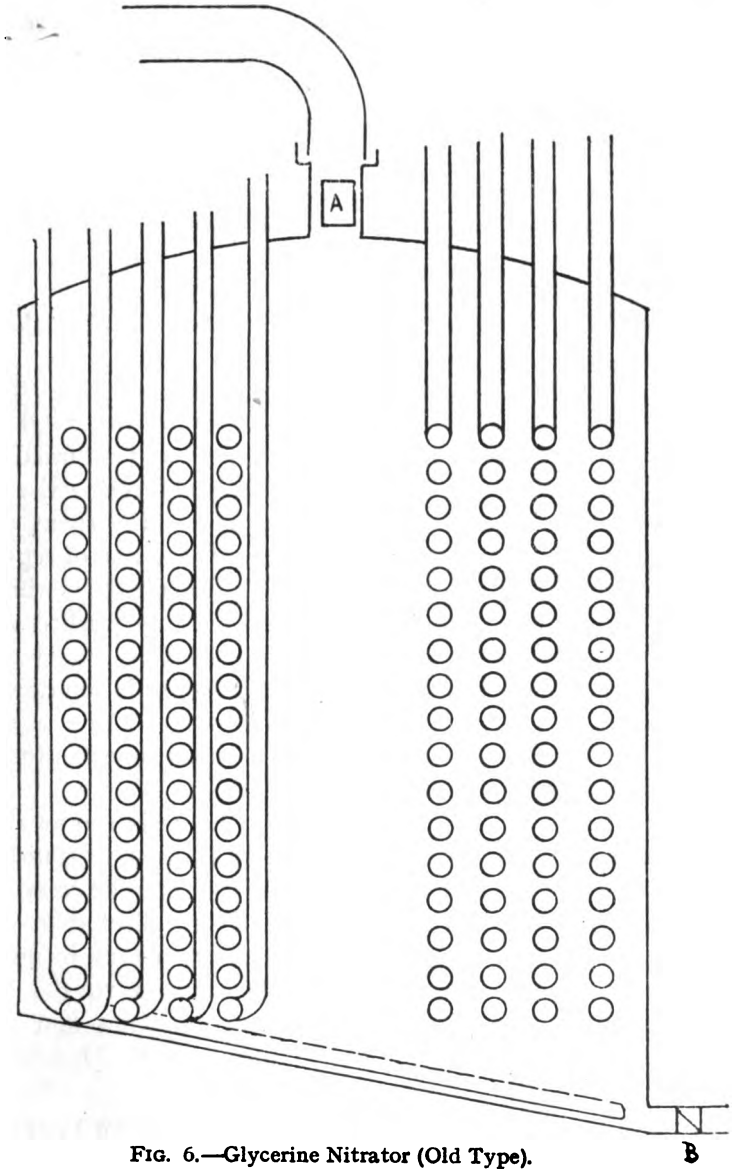

FIG. 6.—Glycerine Nitrator (Old Type).

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measure, as if a suitable quality of glycerine is used and ordinary care taken in nitration no trouble is experienced. When all the glycerine has been added the charge is cooled to 15° C., and then run down a lead-lined gutter to the separating house. The size of charge nitrated varies in different factories from 300 to 1500 lbs. of glycerine. The time taken depends largely on the temperature of the cooling water, and, unless a refrigerating plant is in use, may vary from half an hour in winter to two hours or more in summer. When nitration is finished for the day the nitrators are carefully washed out and allowed to stand overnight with water pressure on the coils. This latter is a precautionary measure to detect any small leakage. The nitrators require to be dismantled about every two months for examination and repairs.

Separating House.—The charge from the nitrating house is received in lead tanks (Fig. 7) and allowed to stand, so that the specifically lighter nitroglycerine rises to the surface. When separation is complete the spent acid is run off from the bottom cock, A, until the line of separation is seen through the window B. The nitroglycerine is then run into the pre-wash tanks and roughly washed with water or dilute soda solution. Being heavier than water, it forms the lower layer, and after pre-washing is run down a gutter to the wash house or filter house. These gutters must be jacketed with warm water in winter time to prevent the nitroglycerine freezing.

The waste acid from the separating house is run away to the after-separating house, where it is allowed to stand for two or three days in order that separation may be complete. The oil that separates is skimmed off or collected by displacement, and carried back to the pre-wash tank in gutta-percha buckets. The waste acid is pumped to the acid department, where it is denitrated with steam and then concentrated. As a rule it contains 10–12 per cent. of nitric acid. To prevent any further separation of nitroglycerine it is advisable to dilute it with one or two per cent. of water, and not to allow the waste acid in the store tanks

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to become too chilled in cold weather. In any case it should be denitrated as soon as possible.

Both separating and after-separating tanks must be provided with thermometers and with compressed air agitators and cooling coils, so that the contents can be mixed and cooled in case of any undue rise in temperature due to decomposition setting in. They must also be provided with drowning tanks placed outside the building.

The necessity for after-separation can be avoided by adding 3–5 per cent. of water to the waste acid. This dissolves the globules of nitroglycerine and prevents the formation of further quantities from unchanged glycerine or glycerine sulphate.

In order that separation may take place in a reasonable time, the mixed acid used for nitration should be allowed to stand for several days before use in order that suspended matter, such as flue dust, iron sulphate, lead sulphate, etc., may settle out. The addition of sodium fluoride to destroy finely divided silica has been patented (A.P. 804,817), and it is claimed that a great saving of time is effected by its use, only about ·001 per cent. being required. On the other hand, patents have been taken out for hastening separation by the addition of siliceous matter such as china clay (E.P. 14,586[12]. Pat. Anm. 38,595 (1911)).

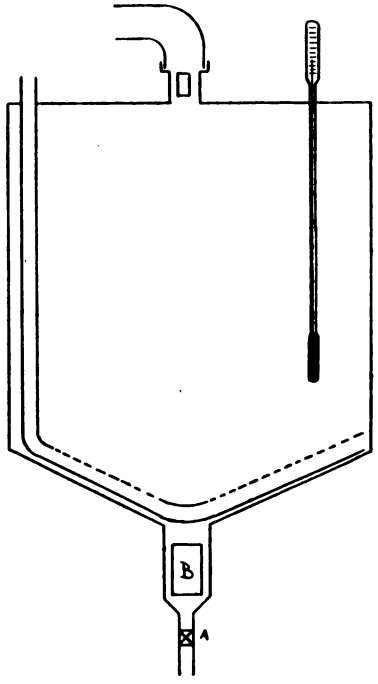

FIG. 7.—Separator for Nitroglycerine.

Wash and Filter House.—The construction of wash tanks varies somewhat in different factories, but the best

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type is that shown in Fig. 8, as this avoids the use of cocks. The oil is agitated with the water or soda solution by means of compressed air and then allowed to settle. The supernatant water is then drawn off by means of the soft rubber hose, which is slowly lowered. The oil is left in the tank and again agitated with water or soda solution, and again separated as before. When finally neutral the oil is drawn off by lowering the outside soft rubber hose. In

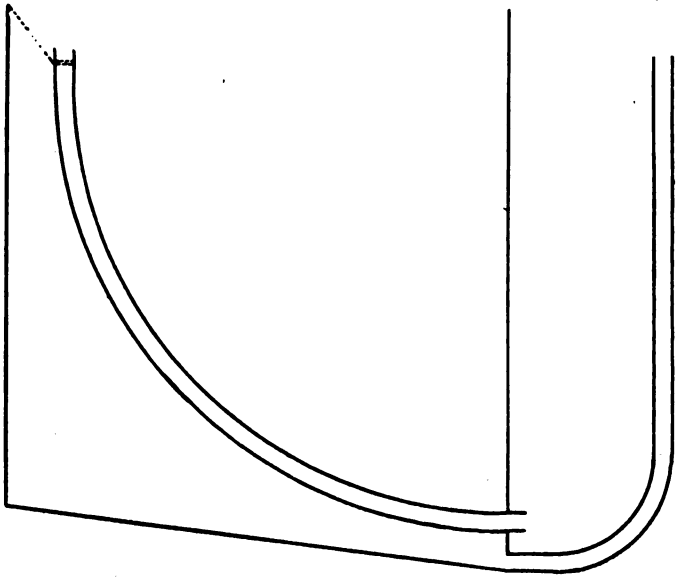

FIG. 8.—Wash Tank for Nitroglycerine.

another type of wash tank the lower layer of oil is separated from the upper aqueous layer, and forced into another tank on the same level by adding water.

The number of washings required is usually nine, viz. three water washings and one soda washing in the pre-wash tanks, and three soda washings and two water washings in the wash house, but washing must be continued until a satisfactory heat test (over ten minutes) is obtained (Section IX.). The strength of the soda solution used is usually about 3 per cent., and all water must be at about 22° C. It is a good plan to do the final washing with distilled water,

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an ample supply of which is usually available at no cost from the condensed steam used in the various drying stoves. Otherwise softened water must be used.

All wash waters both from the pre-wash tanks and from the wash house are led to the "deposit of washings." They are here made to circulate slowly through labyrinths of lead in such a manner that they pass alternately over and under the partitions. This causes them to deposit any globules of oil that they may hold in suspension. The oil thus deposited is collected from time to time, and carried back in gutta-percha buckets to the wash house.

After washing until neutral the nitroglycerine is filtered through sponges sewn up in flannel bags, in order to remove flocculent matter and suspended water. It is then stored in lead tanks in carefully warmed houses, but is almost invariably used within 36 hours of being made.

Nitrator - Separator. — This form of nitrator was patented by Thomson, Nathan, and Rintoul (E.P. 15,983[01], 3020[03]), and is described in the *Journal of the Society of Chemical Industry*, 1908, page 193. The nitrator is shown in Figs. 9 and 10, and the charge is worked as follows. The mixed acid is introduced and nitration carried out as described on page 32. After nitration, however, instead of running the whole charge into a separate vessel for separation, it is allowed to stand for a few minutes, and waste acid from a previous charge then slowly introduced through the pipe and cock C. The nitroglycerine overflows by B into the pre-wash tank situated in the same building. The line of demarcation of the mixed acid and oil is watched through the window A, and the rate of entrance of spent acid governed so that this remains stationary, the nitroglycerine overflowing to the wash tank as rapidly as it separates. When separation is complete the nitrator is left standing full of acid until required for further use. Any further nitroglycerine that has separated is then removed and the waste acid run out by the cock D to the after-separators, if these are used, but when the nitrator-separator is used it is customary to add sufficient water to

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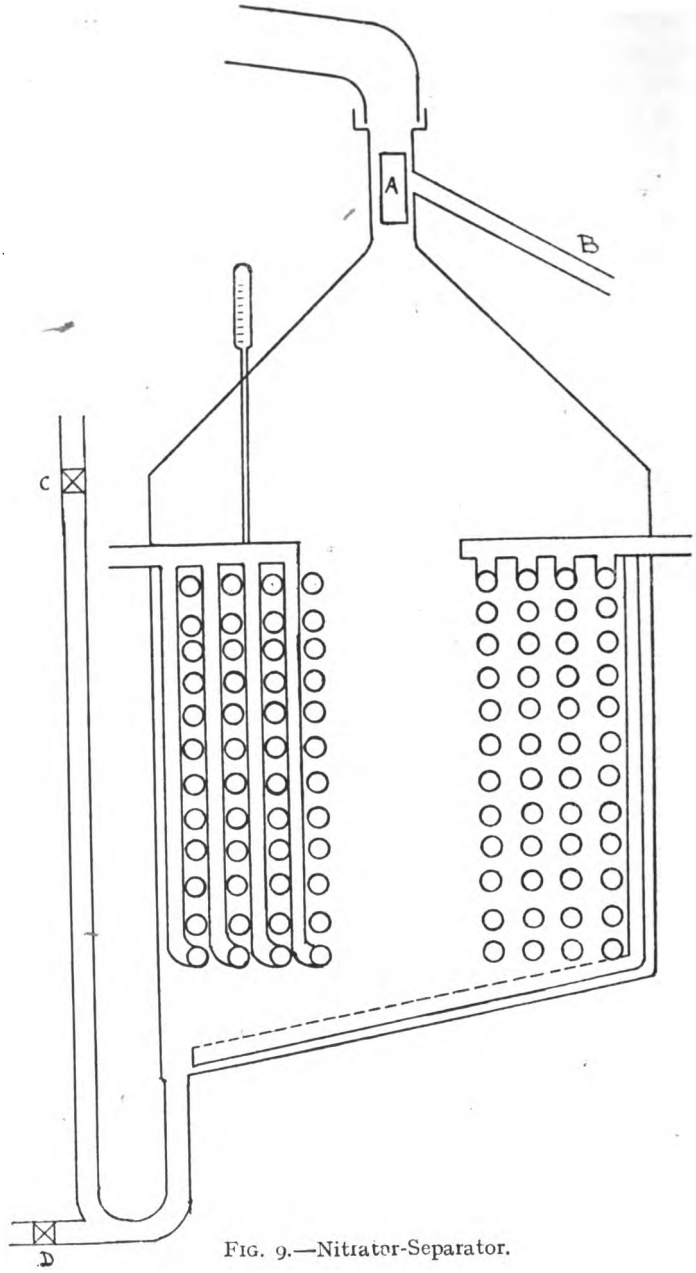

FIG. 9.—Nitrator-Separator.

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the spent acid to prevent any further separation of nitroglycerine, and thus avoid after-separation. This type of nitrator has now been adopted by all the leading works in this country, as it has the following advantages :—

(1) No cocks are used, so that all danger of an explosion being caused by the friction of turning a cock is avoided.

(2) The nitroglycerine is removed as soon as separated, so that the duration of time in which it is in contact with acid is reduced to a minimum.

(3) Repairs and renewals are much less. The older type of nitrator has to be washed out every day after use, and the dilution of the acid which remains adhering to the lead sets up serious corrosion. It is for this reason that the nitrator has to be dismantled every two months, although many authorities state that the corrosion is due to fume. For a matter of fact the lead of the older type of nitrator is exposed to fume no more than the lead of the nitrator-separator. The nitrator-separator, on the contrary, remains full of acid when not in use, so that corrosion caused by the dilution of residual acid is avoided. The first nitrator-separator used at Waltham Abbey ran for 2¾ years without being dismantled, and was then found to be in perfectly good condition, no repairs of any sort being required.

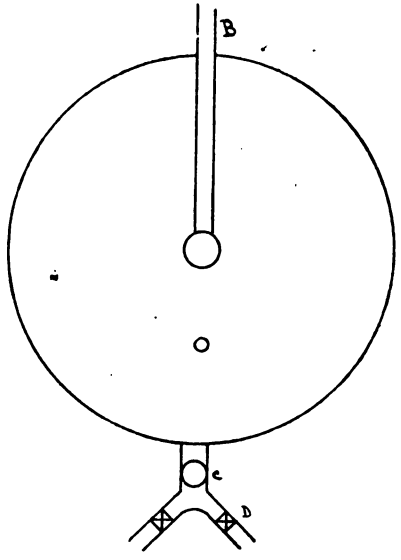

FIG. 10.—Nitrator-Separator. (Plan showing acid inlets and outlets.)

Nitroglycerine is a colourless oil which freezes at 13·3° C., but exhibits the phenomenon of super-cooling to a considerable extent. When in the frozen state it is less easily detonated, and in small quantities is less sensitive to shock,

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although in bulk it is probably more sensitive when frozen than when liquid.

Nitroglycerine has a marked physiological effect, dilating the blood-vessels and causing violent headache and sickness. It is rapidly absorbed through the skin and the majority of workers suffer severely at first, although they usually become immune after a few days. The best remedy for N.G. headache is strong black coffee or aspirin.

Nitroglycerine is very sensitive to shock and is exploded by a kilogram weight falling 4 cm., the nitroglycerine being confined between hardened steel surfaces.

On heating, nitroglycerine decomposes slowly below 100° C. and explodes at about 150° C.

On the manufacturing scale 100 lbs. of dynamite glycerine yields from 230 to 235 lbs. of nitroglycerine. The cost of manufacture in 1914 was about 6*d.* per lb. The cost of manufacture at two Government factories during the period January–June, 1918, was just under £100 per ton, the glycerine accounting for about £40 and the nitric acid for about £45 (*C.T.J.*, 1919, p. 139).

Attempts have been made to use dinitroglycerine mixed with glycerine in order to obtain an explosive that does not freeze so readily, but not much success has been achieved, as the dinitric ester is soluble in water and forms cryohydrates (D.R.P. 17,575[11], 181,385). Much greater success has met the use of dinitrochlorhydrin. This is made by nitrating monochlorhydrin in exactly the same way that glycerine is nitrated. As, however, it is almost solely used in conjunction with nitroglycerine, it is more usual to nitrate a mixture of glycerine and monochlorhydrin (*S.S.*, 1906, 227).

Dinitrochlorhydrin boils with slight decomposition at 190° C., and, although readily detonated by fulminate, is so insensitive to shock that it is not exploded by a 2 kg. weight falling 2 metres. It can only be frozen with great difficulty, and remains liquid at −30° C.

Non-freezing oils have also been obtained by nitrating mixtures of glycerine and diglycerine $(CH_2OHCHOHCH_2)_2O$, this latter being formed by heating glycerine to 290° C.

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with or without a trace of alkali (*S.S.*, 1906, 231 ; E.P. 9572[08]) or at 250° C. in a current of inert gas (E.P. 24,608[10]).

The use of dinitroacetyl glycerine and dinitroformin are mentioned in D.R.P. 209, 943.

Finally, dinitroglycol has been used in France. It is a more powerful explosive than nitroglycerine, and it is claimed that it can be manufactured at a lower cost (*P.S.*, **16**, 72). It does not freeze above −20° C.

Interesting results have been obtained from a study of the solubility of nitroglycerine in sulphuric acid of various strengths, and in sulphuric acid containing nitric acid. The solubility is due to two causes, viz. the true solubility of nitroglycerine as such and the solubility due to the decomposition of the explosive. With sulphuric acid alone the solubility rises slowly with increasing strength of acid until an acid strength of about 50 per cent. is reached. Then the rise in solubility becomes very rapid, the curve being almost vertical in the region where H_2SO_4=90 per cent. Between 50 and 90 per cent. great instability is exhibited, and the nitroglycerine is liable to undergo spontaneous decomposition with uncontrollable violence, so that manufacturing operations must be so adjusted that nitroglycerine is not brought into contact with acids of this strength.

With sulphuric acid containing water and nitric acid in the proportion 1 : 1·1 the case is somewhat different, and instability is most marked when the mixture contains under 60 per cent. of sulphuric acid, *i.e.* contains more than 40 per cent. of nitric acid of 52 per cent. strength.

The curve of total nitroglycerine absorbed in acids containing varying percentages of sulphuric acid shows a very marked peak where H_2SO_4=50, and becomes almost vertical above the point H_2SO_4=80. The amount of nitroglycerine dissolved as such also becomes very steep when H_2SO_4=80, but the peak where H_2SO_4=50 is only very slight, showing that the solubility is chiefly due to decomposition. These values are important both as a guide to suitable acid mixtures for obtaining a good yield and also as showing that nitroglycerine may become

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dangerous through violent decomposition when in contact with acids of certain strengths.

Glycerine is invariably obtained from natural sources, viz. from fatty oils, and is a by-product from the soap industry. Over 50 per cent. of the world's production of glycerine is used in the manufacture of explosives, the remainder being used chiefly for medicinal purposes. In view of the extremely simple chemical structure of the glycerine molecule, it is curious that no synthesis has yet been devised that is even remotely suited for use on a large scale. Proposals have been made to carry out the synthesis by condensing acetylene with methane in the presence of suitable catalysts, and then converting the butylene thus obtained into glycerine by treatment with chlorine and alakli—

$$\begin{array}{l} CH \\ \vdots\!\vdots\!\vdots \\ CH \end{array} + CH_4 \rightarrow \begin{array}{l} CH_2 \\ | \\ CH \\ \| \\ CH_2 \end{array} \rightarrow \begin{array}{l} CH_2Cl \\ | \\ CHCl \\ | \\ CH_2Cl \end{array} \rightarrow \begin{array}{l} CH_2OH \\ | \\ CHOH \\ | \\ CH_2OH \end{array}$$

but no success has been achieved, as the reactions are not so simple as they appear.

Glycerine is always formed as a by-product in the fermentation of sugar by yeast, and is probably formed by the auto-digestion of the yeast cell. A process based on fermentation would therefore not seem to be impossible, and the United States Government claim that they have evolved such a process and have proved it to be feasible on a manufacturing scale. No details have been made public, but the process has been communicated to the Allied Governments.

Prior to the war the price of "dynamite glycerine" was variable, but in the neighbourhood of £100 per ton, about 10½*d.* per lb., and although this would no doubt come down considerably with increased supplies, still the demand is so great that a sufficient margin would remain to allow a good profit on a synthetic process should one be discovered.

Possibly glycerine may be displaced in the future to

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some extent by glycol, as this seems somewhat easier to obtain by synthetic means—

$$\begin{array}{ccccccc} CH & & CH_2 & & CH_2Hlg & & CH_2OH \\ \| & \rightarrow & \| & \rightarrow & | & \rightarrow & | \\ CH & & CH_2 & & CH_2Hlg & & CH_2OH \end{array}$$

The best results are obtained by using bromine, the yields under these circumstances being said to be almost theoretical. The capital cost, however, is very heavy, and the unavoidable mechanical loss which takes place in all manufacturing operations is a serious item when an expensive material such as bromine is involved, so that should glycol be manufactured extensively by this method it would be necessary to use chlorine. This, owing to its lower atomic weight, has the further advantage of requiring considerably less alkali for the final hydrolysis. Dinitroglycol is quite suitable for the manufacture of explosives, and its use is actually authorized in France (see page 41). It contains exactly the right amount of oxygen for its complete oxidation—

$$\begin{array}{l} CH_2ONO_2 \\ | \\ CH_2ONO_2 \end{array} = 2CO_2 + 2H_2O + N_2$$

Nitrocellulose

Cellulose has the formula $(C_6H_{10}O_5)_n$, the molecular weight being quite unknown, although there is no doubt that the molecule is of great complexity. The structure is also quite unknown, but as nitric esters containing 13·96 per cent. of nitrogen can be obtained it is probable that there are three hydroxyl groups reckoned in the simple molecule $C_6H_{10}O_5$, the trinitric ester of which would contain 14·14 per cent. of nitrogen. It may be said at once that these esters with high nitrogen content are unstable and of no commercial value, as it is almost impossible to obtain stable nitrocelluloses containing much over 13·1 per cent. of nitrogen.

Some authorities attempt to classify nitrocelluloses by ascribing the imaginary formula $C_{24}H_{40}O_{20}$ to cellulose, but

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the cellulose esters as at present known are undoubtedly mixtures, and it is much more scientific to characterize them by their nitrogen content and by their solubility in ether-alcohol (one volume ether and two volumes alcohol). Officially in this country guncotton is defined as nitrocellulose containing over 12·3 per cent. of nitrogen, and of which not more than 15 per cent. is soluble in alcohol-ether. Esters containing less than 12·3 per cent. of nitrogen, and more or less completely soluble, are known as collodion or pyroxylin. In factories guncotton is generally known as G.C. and collodion cotton as C.C., whereas N.C. denotes nitrocelluloses in general.

The whole theory and practice of the preparation of nitrocellulose rests on an empirical basis, owing to the complete absence of any knowledge of the structure of the cellulose molecule. Undoubtedly all forms of cellulose are colloidal in nature, but to what extent the substance known as cellulose represents a pure compound we have no means of ascertaining. Probably there are numerous forms of cellulose, but speculation on this line must remain guesswork in our present state of knowledge. In the manufacture of nitrocelluloses the chief raw materials are cotton waste, used chiefly for making guncotton and collodions for varnish or celluloid, cotton wadding used for collodion for blasting explosives, paper used for various purposes such as celluloid, and purified wood or wood pulp used for certain smokeless powders. It is probable that great progress has been made in the use of wood in Germany owing to their military requirements and the failure of the cotton supply through the Allied blockade, but no information on this point is as yet forthcoming.

Cotton waste used for making guncotton undergoes several processes of purification before it is fit for nitration. These consist in extracting with solvents and scouring with soda to remove oil, washing and bleaching. This preliminary treatment is carried out by the suppliers and not by the explosives works. As delivered, it should contain under 8 per cent. of moisture and not more than ·4 per cent. of oil. It

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should not be harsh to the touch or friable (overbleaching) and should only produce slight reduction when boiled with Fehling's solution (oxycellulose). It is picked over by hand to remove bits of string, nails, etc., and then run through a teasing machine to loosen knots, dust being at the same time removed by a fan. After drying, to reduce the moisture to 1 per cent. or less, it is ready for nitration.

Guncotton.—The mixed acid used for manufacturing guncotton in this country (13 per cent. of nitrogen) has the composition—

HNO_3	23
H_2SO_4	70
H_2O	7

In the old *Abel process* the cotton in lots of 1¼ lbs. was dipped into the mixed acid contained in cast-iron pans. After five minutes it was lifted out, squeezed by hand on an iron grating and then placed in stoneware pots set in a trough of running water. Nitration proceeded for 12 hours, after which time the excess of acid was wrung out as far as possible in hydro-extractors and the guncotton drowned quickly in a large volume of cold water. This process was later modified into the *direct dipping process*, in which the cotton is digested with the mixed acid for from 8 to 24 hours at 20° C. in water-cooled iron pans, the ratio of acid to cotton being 6 : 1. Both these processes, which are still in use on the Continent and in some collodion works in this country, were wasteful in acid and injurious to the workmen owing to the fumes. In addition, a great deal of labour was involved and charges fuming off were frequent. Indeed in the Abel process a drop of perspiration falling on to a charge when digesting was frequently sufficient to cause it to fume off. These disadvantages led to the introduction of *the displacement process* by Nathan and Thomson (E.P. 8278[03]. *A.E.*, 1906, 77), by which great economies in material and labour were effected, all fumes avoided, and a superior product obtained. The process has been very generally adopted by explosives works in this country and

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is coming into extensive use on the Continent and in America. Up to the present it has not been generally used for the manufacture of collodion, but by selecting a suitable mixed acid there would seem no good reason why it should not be used for this purpose, and no doubt the extensive guncotton plants that have been erected for war

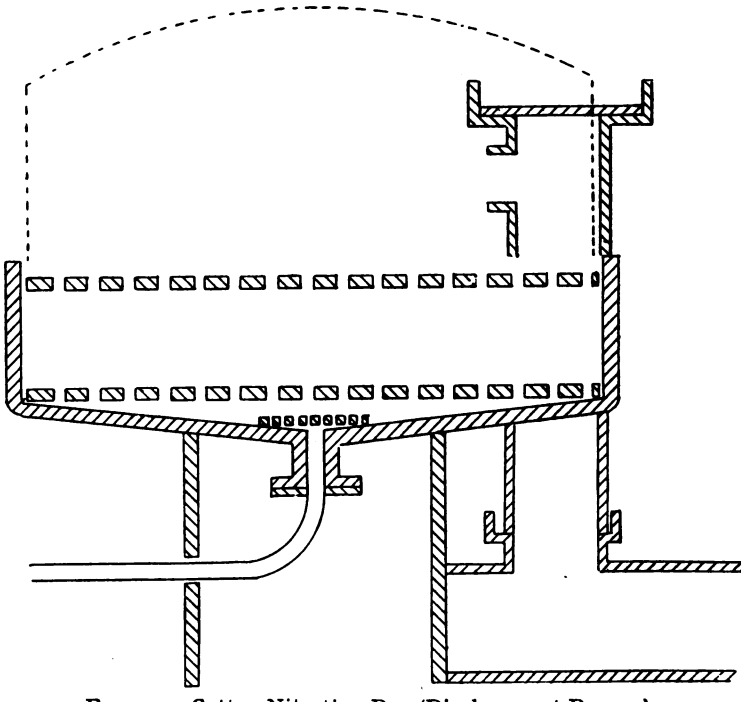

FIG. 11.—Cotton Nitrating Pan (Displacement Process).

purposes will be diverted in this direction, the collodion being used for medicinal and photographic purposes, artificial silk, celluloid, varnish, aeroplane dope, etc.

The nitrator is shown in Fig. 11, and consists of a shallow earthenware pan, 3 ft. 6 in. diameter, 10 in. deep at the sides and 12 in. deep at the run-off cock at the centre. It has a false bottom of perforated stoneware segments. The run-off cock is connected with the mixed acid store tanks, waste acid tanks, and with a drain, and four nitrators are usually

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grouped together so as to be worked as one unit. In carrying out a nitration mixed acid (650 lbs.) is allowed to run into each pan, and then 20 lbs. of cotton waste added in single handfuls and quickly pushed under the surface of the acid by means of an aluminium fork. During this process the pan is covered by a light portable aluminium hood connected with a fume pipe so that all acid fume is drawn off. When the whole of the cotton has been added it is covered with loose perforated stoneware segments, on to the surface of which a layer of chilled water at 5° C. is run *slowly*. This seals the plant and prevents any escape of fume. Nitration takes 2½ hours, and the fuming off of a charge is almost unknown. When nitration is complete the waste acid is run off at the bottom at the rate of about one gallon per minute, and at the same time and at the same speed cold water is run on to the top perforated plates. Water being a great deal lighter than the waste acid, the line of demarcation between them remains fairly sharp and about 500 lbs. of waste acid are recovered in a state fit for revivifying with nitric acid and oleum. This portion of the waste acid has the composition—

HNO_3	18
H_2SO_4	72·7
H_2O	9·3

the rest is somewhat weaker—

HNO_3	18
H_2SO_4	61
H_2O	21

and is denitrated and concentrated. Displacement takes about three hours, and the loss of acid is only about 1 per cent. of the weight of the guncotton made. The yield of guncotton is about 170 per cent. Up to the present these displacement nitrators have always been constructed of the dimensions given, as stoneware vessels of large size are expensive and not very satisfactory. There would seem to be no reason, however, why larger vessels should not be built of cast iron lined with acid-proof stoneware segments,

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of enamelled iron or of ferro-silicon. Naked cast iron or lead cannot be used, as, although they would be unattacked during nitration, they would be seriously affected by the dilution of the acid during the later stages of the displacement.

The *centrifugal nitrating process* is used extensively in Germany and also to some extent in this country, especially for the mauufacture of collodion cotton for blasting explosives. In this case nitration is carried on in a centrifugal

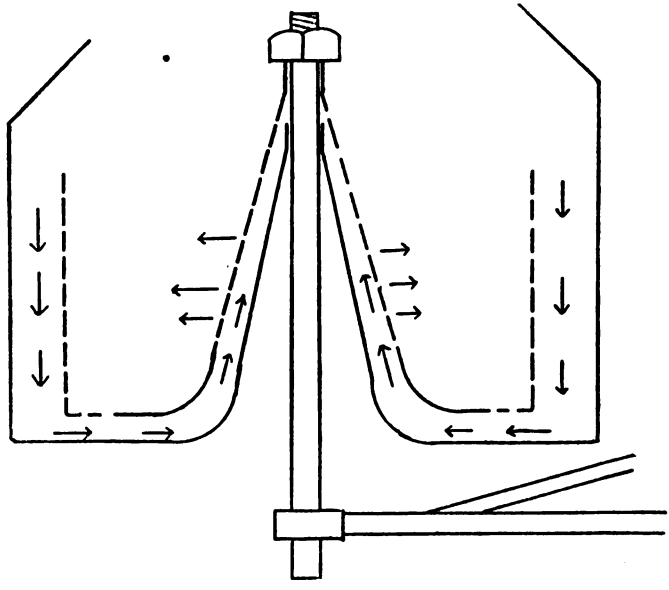

FIG. 12.—Nitrating Centrifugal.

machine so arranged that the rotation of the basket causes the acid to circulate through the cotton. A simple type of nitrating centrifugal is shown diagrammatically in Fig. 12, the circulation of the acid being indicated by the arrows. It will be noticed that the vertical spindle passes through the central conical part of the outer case by a sleeve situated well above the acid level, and is attached to the basket at the top of the central perforated cone. During nitration the machine rotates very slowly, but when nitration is complete the excess of acid is run off and the machine speeded

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up so as to wring out the superfluous acid. This wringing, however, must not be carried too far or there will be considerable danger of the charge fuming off, and this applies to all processes in which the excess of acid is removed by whizzing. As a rule the acid content of the nitrated cotton is not reduced below 50–60 per cent. before drowning. The charge for a centrifugal machine is 15–25 lbs. of cotton and 750–1250 lbs. mixed acid.

The composition of the acid used for making collodion depends on the duration and temperature of nitration, process of nitration employed, and on the nature of the product required. The viscosity of the solutions of the collodion depends very largely on the temperature at which nitration takes place. For blasting explosives high viscosity is required, so that nitration is carried out at or about the ordinary temperature. A suitable mixed acid for this purpose is—

HNO_3	25·0
H_2SO_4	57·5
H_2O	17·5

These collodions usually contain about 12 per cent. of nitrogen.

For smokeless powders the viscosity is much less important, nitrogen content, which is a measure of power, being the important point. No reliable information is available as to the acids used for nitrating cellulose for sporting powders, but the United States military powder is made with an acid of this composition—

HNO_3	21·5
H_2SO_4	63·5
H_2O	15·0

Nitration is carried out by the displacement process at a temperature of 30°C. and takes from 1 to 2½ hours. Seven hundred pounds of acid are used for 20 lbs. of cotton, and the product contains about 12·7 per cent. of nitrogen and is almost completely soluble in alcohol-ether. A similar nitrocellulose is used by the French Army. The yield is 155 per

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cent. For varnishes and photographic films low viscosity is required, and hence nitration is carried out at a higher temperature, although this usually entails a lower yield. A good low grade pyroxylin can be made with an acid—

HNO_3	17
H_2SO_4	55
H_2O	28

nitrating for 15 minutes at 45°–55° C. and using 50 parts of acid to each part of cotton. The product contains about 12·4 per cent. of nitrogen, and the yield is about 130 per cent.

Washing.—After a preliminary wash to remove the greater part of the acid the nitro-cotton is boiled several times with water. This boiling is carried out in pitch pine vats with a run-off cock below a perforated false bottom, heating being effected by direct steam. The number and duration of the boilings varies at different factories, but good results are obtained by two boils of twelve hours each, followed by five of four hours each and then three of two hours each, the object of the long boils at first being to hydrolyse unstable esters. Collodion cotton for blasting explosives will not stand such drastic treatment, as boiling spoils the viscosity of the jelly formed with nitroglycerine. Hence it is usual only to wash it several times with water near the boiling point. Nitro-cotton cannot be rendered stable by boiling alone, as acid gets into the hollow fibres and cannot be removed by this process. The only way to remove this is by the pulping process introduced by Abel. After boiling, therefore, the cotton is removed to pulpers and made to circulate between rapidly revolving knives until reduced to a fine pulp. The construction of a pulper is shown diagrammatically in Fig. 13. Here again the process must not be carried too far with collodion intended for blasting explosives, or the gelatinizing properties will be spoilt. A large amount of water is used during the pulping operation, and when pulping is complete the creamy liquid is pumped into tanks at the top of the building and then made to flow along long wooden gutters

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lined with woolly blanket material. The woolly material collects a large amount of grit, and particles of iron are collected by electro-magnets placed at intervals. The pulp then enters a large timber vat, known as a poacher, fitted with a wooden paddle, where it is allowed to settle and the supernatant milky water drawn off. The pulp is then stirred up with more water and again allowed to settle. It is usually given three washes in the poacher, and is then removed to hydro extractors and as much of the water as possible extracted. As obtained from the extractors it contains 25–35 per cent. of moisture, and in this state is perfectly safe.

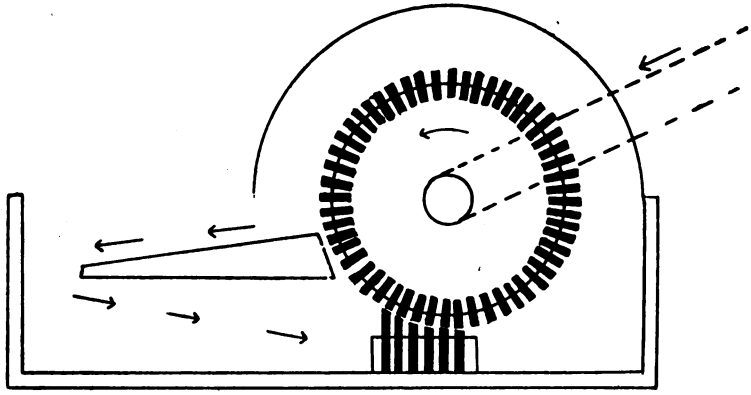

FIG. 13.—Pulping Machine for Nitrocellulose.

Dry nitrocellulose, however, is very easily inflamed, and all operations with it must be carried out in danger buildings. The stoves are light timber structures surrounded by earth mounds and certain movable trays on which the nitrocellulose is spread. These trays may be constructed of light timber frames with canvas bottoms, but fine copper gauze or zinc sheet is much better. They are about 2 in. deep and must be earthed, as dry nitrocellulose is very easily electrified. Heating is brought about by blowing in warm air, but the temperature of the stove must not exceed 40° C. To avoid nitrocellulose dust being blown about, some firms compress the wet cotton into slabs by means of hydraulic presses before drying. This system has many advantages,

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although drying takes somewhat longer. Only moderate pressure is used in forming the slabs so that they are easily broken up when dry. In any case, as dry nitrocellulose is much more dangerous when warm, the stoves must not be unloaded until they have cooled.

If the nitrocellulose is going to be used for making smokeless powder by gelatinization with alcohol and ether, or if it is going to be used for making a solution in which alcohol is employed as a solvent, there is no need to dry it. Under these circumstances it is packed into cylinders and alcohol then forced through it in a downward direction. The first runnings are pure water, followed by dilute spirit which is afterwards rectified. The last runnings are stronger spirit, and are used for the first washing of the next batch. The process is very similar in principle to the displacement process of nitrating, and is generally known as "alcoholizing." The average cost of manufacturing guncotton (13 per cent. nitrogen) at three Government factories during the period January–June, 1918, was £137·8 per long ton, about 1*s.* 3*d.* per lb., this amount being made up as follows: cotton waste £50·6, nitric acid £44·5, labour, etc., £36·6, sulphuric acid £6·1 (*C.T.J.*, 1919, p. 139).

Nitroaromatic Compounds

Dinitrobenzole.—Although mononitrobenzole cannot be exploded either by a blow or by a fulminate detonator, the dinitro-compound is sufficiently unstable to be exploded by either of these means, although only with difficulty, explosion being brought about by a two-kilogram weight falling 200 cm., the dinitrobenzole being confined between hardened steel surfaces. It is made by nitrating nitrobenzole with mixed acids at 100° C., its manufacture being fully described in the volume on "Coal Tar Dyes and Intermediates" in this series. The pure product melts at 91° C., but the commercial article at 80°–85° C.

Dinitrotoluol and Trinitrotoluol.—Both these are used extensively in the manufacture of explosives, especially the

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latter. Trinitrotoluol (T.N.T.) is exploded by a two-kilogram weight falling 108 cm., and is fairly readily exploded by fulminate of mercury. In the pure state it is used for naval and military purposes in torpedo charges and to a lesser extent in shells. It finds its chief application, however, when mixed with nitrates, especially ammonium nitrate, for shell filling (amatol) and as a blasting explosive. Although T.N.T. can be obtained from toluol in one step this process is very extravagant in acid, and consequently the manufacture is invariably carried out in two or three steps, depending to some extent on the quality of product desired and on what other products are being manufactured simultaneously. In spite of the enormous output of T.N.T. for war purposes, it is doubtful if the most economical process for use under peace conditions has yet been evolved. This is due to the shortage and excessive price of sulphuric acid having compelled manufacturers to work more with a view to economy of acid and oleum than with a view to minimum cost of production. Under present circumstances only about 2 lbs. of T.N.T. are obtained from 1 lb. of toluol, this corresponding to a yield of about 77 per cent. of theory. With increased supplies of oleum available it will probably be found more economical to complete the final nitration with a stronger acid and a correspondingly lower temperature, as by this means better yields will be obtained.

In carrying out the nitration in two steps, toluol is first nitrated to mononitrotoluol * (M.N.T.) and this then further nitrated to T.N.T. The composition of the mixed acid used for the final nitration depends on what acids are available for mixing, but both the following give good results. The latter, however, usually requires a small amount of oleum—

HNO_3	16	18
H_2SO_4	78	78
H_2O	6	4

The amount of mixed acid taken is such that it contains

* For details see Barnett, "Coal Tar Dyes and Intermediates," in this series, p. 16.

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1¾ lbs. of nitric acid for every pound of M.N.T. to be nitrated. The size of the charge varies in different works, but by using

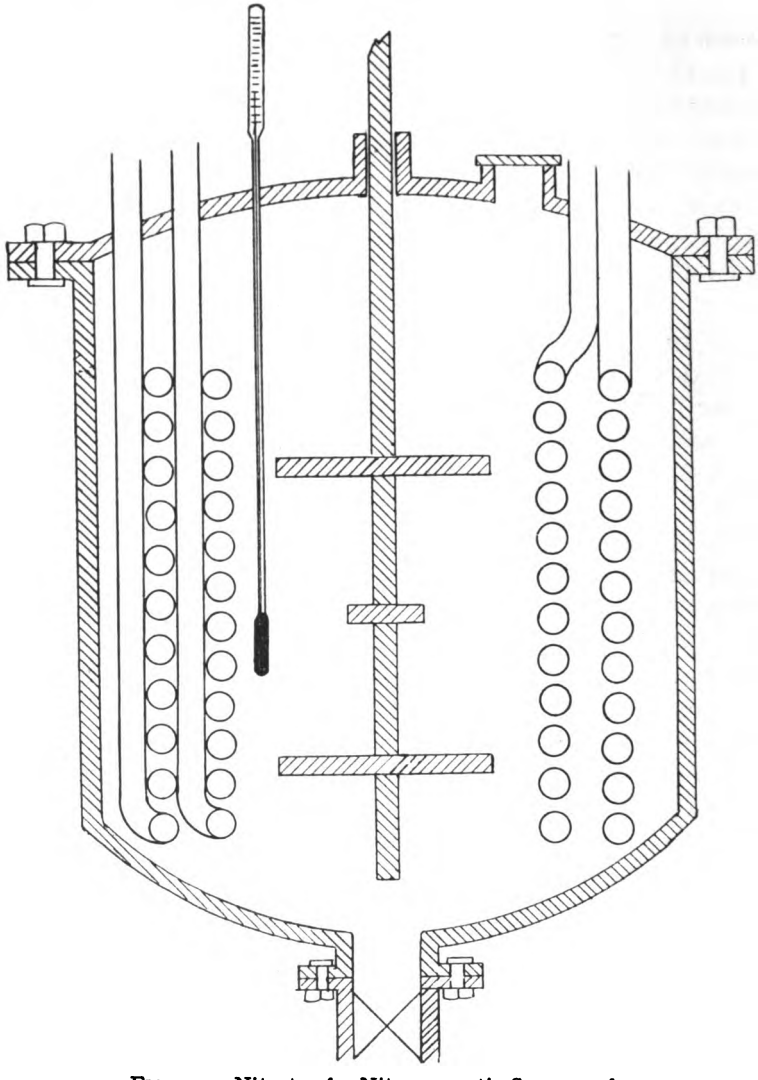

FIG. 14.—Nitrator for Nitroaromatic Compounds

12,000 lbs. of mixed acid about 2000 lbs. of T.N.T. can be made, and this can be worked conveniently in a nitrator of

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1000 gals. working capacity. These nitrators are made of cast iron with internal lead coils for heating and cooling, and are provided with an efficient mechanical agitator (Fig. 14). In working a charge the mixed acid is first run in and then the M.N.T. During the addition of the latter the temperature is not allowed to rise above 50° C. When all the M.N.T. has been added the temperature is raised at the rate of about 1° C. every two minutes. At about 70° C. there is considerable evolution of heat, and care must be taken to check this by turning water into the coils. The charge is generally cooked for 1 hour at 100° C., and the temperature then slowly raised to 120° C. and maintained at this point until nitration is complete. This generally takes about ½ hour, and is ascertained by taking a sample and determining the setting point. The charge is then cooled to 85° C., and about ¾ gallon of water for every hundred pounds of mixed acid used run in. The object of this is to reduce the solubility of the T.N.T. in the spent acid (D.R.P. 254,754). During the addition of the water the heating up of the charge must be carefully checked and the temperature not allowed to rise above 100° C. The charge is then again cooled to 85° C., the agitator stopped and separation allowed to take place for an hour. The lower layer of spent acid is then drawn off and the supernatant T.N.T. run off into a tank of boiling water for washing. It must be washed free of acid, and on no account must any alkali be used, as if so the T.N.T. becomes very sensitive. Usually four washes suffice to remove all the acid, after which the T.N.T. is dried by heating to 105° C. and then either cast in shallow trays or flaked on an internally water-cooled revolving drum. Instead of adding water to the charge after nitration, dilute sulphuric acid obtained from the vitriol concentrating plant can be conveniently used.

The waste acid from the trinitration contains considerable quantities of T.N.T. in solution. This can be recovered by agitating it at 80° C. with the M.N.T. to be used for the next charge, in which case any nitric acid present is used up with the formation of a small quantity of dinitro-

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toluol. In this case the final waste acid will contain about 3 per cent. of nitrous acid, and is denitrated and concentrated for further use. In some works, however, the T.N.T. spent acid is revivified by the addition of nitric acid and then used for making M.N.T. The T.N.T. made by this process has a setting point of about 76° C.

The three-stage process is somewhat more complicated, but is frequently used by those works which make *m*-toluylenediamine. As before, the toluol is first nitrated to M.N.T. This is then converted into the dinitro-compound by treatment with an acid of the composition—

HNO_3	30
H_2SO_4	64
H_2O	6

nitration being carried on at 60° C. and the charge finally cooked at 80° C. The dinitro-compound can then be directly nitrated to T.N.T., or it can be roughly separated into fractions of high and low setting point. This is done by draining in on fine mesh wire gauze trays at a temperature near its setting point, when most of the lower melting fractions run off, the last being got rid of by whizzing. If the residue is then nitrated, T.N.T. with a setting point of 80° C. is obtained. The oil can also be nitrated, and for this purpose Escales recommends an acid of the composition—

HNO_3	10·2
H_2SO_4	82·2
H_2O	7·6

allowing a ratio of acid to dinitro-compound of 2·8 : 1. The setting point of the T.N.T. thus obtained is 72°–74° C. Those qualities of T.N.T. that have low setting points are obviously mixtures of various isomeric trinitrotoluenes with some dinitrotoluene.

For the manufacture of blasting explosives the setting point of T.N.T. is not a matter of importance, but for detonating fuze and certain military purposes in which the explosive is to be used unmixed with nitrate it is necessary to employ almost pure *sym*-trinitrotoluene (M.P. 81·9) in

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order to secure complete detonation. For this purpose the crude T.N.T. can be crystallized from alcohol containing 20 per cent. of benzole, from *o*-nitrotoluol (E.P. 17,003[14]) or from sulphuric acid monohydrate (D.R.P. 237,738). The setting point can also be raised by washing with alcohol or boiling sodium sulphite solution, or by allowing the crude product to drain on wire gauze in stoves maintained at a temperature approximating to its setting point.

When organic solvents are employed in its purification the residue left on distillation consists of a mixture of di- and tri-nitrotoluols. On nitration it yields a product which remains liquid at the ordinary temperature and is much used for manufacturing blasting explosives, especially non-freezing jellies, as it mixes very readily with nitroglycerine. The first liquid product of this nature was manufactured by J. W. Leitch & Co., of Huddersfield, and sold as "liquid trinitrotoluol." The method of manufacture is a trade secret, and although other firms have produced similar liquids, they have not the same capacity for remaining fluid and usually become pasty on standing. Trinitrotoluol, like all other aromatic nitro-compounds, has a decidedly toxic effect on those working with it. The preliminary symptom is a rash which appears chiefly on the arms and may be followed by toxic jaundice, several fatalities having occurred from this cause. The trouble is most marked during the summer months, and some individuals are much more sensitive than others. Those that are sensitive should not be allowed to continue working, and any worker who shows signs of rash should at once be given a change of work until these symptoms have subsided. In Great Britain there is a statutory obligation to provide baths, special clothes, and rubber gloves for those handling T.N.T. and kindred compounds. As regards rubber gloves, the author's experience is that they do more harm than good, as the interior gets soiled when taking off, and thereafter the sweating of the hands that they induce causes T.N.T. to be more readily absorbed through the skin. Closely woven cotton gloves or chamois leather gloves are more satisfactory, as they can be washed

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after each day's work if necessary. Probably the best preventive methods will be found to lie in ample ventilation, prevention of dust, and the artificial cooling of the work-rooms during warm weather. When possible, workers should be given a change of employment at regular intervals. Under the provisions of the Factory Act they must be medically examined at frequent intervals.

Trinitrotoluol is known under various trade names such as trotyl, tritol, triolite, but most usually by the initials T.N.T. Until recently it was specially exempted from treatment as an explosive by an Order in Council made under the Explosives Act. The numerous accidents that occurred during the war have led to the withdrawal of this exemption, and trinitrotoluol must now be manufactured under "danger" conditions. The average cost of manufacture of crude T.N.T. (setting point 76°–78° C.) at six Government factories during the period January–June, 1918, was £110 per ton, the average consumption of toluol being ·491 ton per ton of T.N.T. These figures must, however, be accepted with some reserve, for whereas toluol, acid, and nitrate seem to be reckoned in long tons (2240 lbs.) it is not stated whether T.N.T. is reckoned in long tons or in short tons (2000 lbs.), the latter being almost invariably employed when dealing with explosives (*C.T.J.*, 1919, p. 117).

Picric Acid (Trinitrophenol).—Under the names of Lyddite, Melinite, etc., picric acid was for many years the standard shell-filling used by almost all the Powers, but during recent years it has largely been replaced by the cheaper, safer, and more convenient T.N.T. The great disadvantage of picric acid lies in its acidic character and its capacity for forming highly sensitive salts, especially with the heavy metals, the lead salt being the most dangerous. Picric acid cannot safely be mixed with nitrates such as ammonium nitrate, as owing to its strongly acid character a certain amount of free nitric acid would be liberated should the explosive become damp. Its use is confined to military purposes, and it will probably be completely displaced by T.N.T. at an early date.

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Picric acid was originally manufactured by direct nitration of phenol, but this process is never carried on now, although in 1900 a process for nitrating phenol in paraffin was described (E.P. 16,628[00]) and good results claimed. The usual process is based on the sulphonation of phenol to the monosulphonic acid with subsequent nitration and simultaneous replacement of the sulphonic acid group. The sulphonation is carried out with 4 parts of sulphuric acid of 98 per cent. strength at 100°–105°. Another four parts of sulphuric acid are then added and the melt run into a nitrator and cooled to 20° C. The nitrator may be made of cast iron, but enamelled iron is preferable. Nitration is brought about by the slow addition of mixed acid composed of equal parts of concentrated sulphuric acid and nitric acid of 61 per cent. strength. At first the temperature is kept below 40° C., but after half the mixed acid has been added the temperature is slowly increased to 70°–80° C. The charge is then run into a series of stoneware pots and diluted with an equal volume of water. On cooling the picric acid crystallizes out and is filtered off, washed, whizzed, and finally dried at a low temperature, usually 35° C.

A more modern process avoids the use of phenol, chlorbenzole being used as the starting out point. This is first nitrated to the dinitro-compound, which on boiling with soda gives dinitrophenol.* This latter is readily nitrated to picric acid, but details of the nitrating process are not available at present. This is probably the best process, and is likely to replace the older process if picric acid continues to find use as an explosive. The cost of manufacture of picric acid at H.M. Factory, Greetland, during the period January–June, 1918, was £184·7 per long ton, about 1*s.* 8*d.* per lb. The process used was the nitration of phenol sulphonic acid (*C.T.J.*, 1919, p. 162).

Picric acid melts at 122·6° C.

Tetranitroaniline.—This was first obtained by Flürsheim in 1910 by nitrating *m*-nitraniline or certain near derivatives

* For details see Barnett, "Coal Tar Dyes and Intermediates," in this series, pages 18 and 66.

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of *m*-nitraniline (E.P. 3224[10]). The patentee describes the nitration of *m*-nitraniline as follows: "One part of meta-nitro-aniline is dissolved in 36 parts of strong sulphuric acid (either concentrated acid or monohydrated or fuming acid) at the ordinary temperature or with external cooling, 2½ parts of sodium nitrate are then added, and the mass is allowed to remain at the ordinary temperature or at a lower or only slightly higher temperature for several days." He also states that the nitration can be carried out by heating to 70° C. and then finally, after the reaction has subsided, to 100° C. It will be seen that a very excessive amount of sulphuric acid is required, and the cost of the process could only be kept within reasonable bounds by using the spent acid for other purposes. The compound has been used for detonator compositions, but tetranitromethyl aniline has been found more suitable for this purpose.

Tetranitromethyl Aniline.—This is really trinitro-methyl phenylnitramine $CH_3N<^{C_6H_2(NO_2)_3}_{NO_2}$, and is known usually as tetryl or tetralite. It is manufactured by nitrating dimethyl aniline, one methyl group being simultaneously oxidized. The process is described by Escales ("Nitro-sprengstoffe") as follows:—

One hundred kilos. of dimethyl aniline are slowly added to 1000 kilos. of well-cooled concentrated sulphuric acid of 97–98 per cent. strength. A pale brown coloured solution is obtained, but it must not be too dark and must remain transparent. As the solution decomposes on standing the nitration should be effected as soon as possible. This is brought about by running the solution slowly into 530 kilos. of nitric acid of 87 per cent. strength which has previously been warmed to 40° C. The strength of the nitric acid is important, as if a stronger acid is used the product separates in large crystals which are difficult to wash. During the addition of the dimethyl aniline solution the temperature at first is not allowed to exceed 44° C., but after two-thirds has been added it is allowed to rise to 55° C., and when the addition is complete the charge is cooked for two hours at

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this temperature and is then cooled and allowed to stand overnight. The nitro-compound crystallizes out and the majority of the spent acid can be run off. On the average this has the composition—

HNO_3	11·05
H_2SO_4	74·04
H_2O	12·09
NO_2	2·58
Nitro-compounds	·24

The nitro-compound is then collected on filters, and after washing first with dilute sulphuric acid and then with water is dried. The yield of the crude product is 210 kg., but it is unstable and should be purified at once by recrystallization from benzole. The benzole is recovered from the liquors by distillation, and to avoid danger of the residue exploding it is best to add a large volume of water before commencing the distillation. These residues are quite valueless and are burnt.

Tetryl melts at 129°–130° C. and is very poisonous. Up to the present it has only found application for detonators or shell fuzes, but its use as a shell filling or as an ingredient in blasting explosives has been proposed. During the period January–June, 1918, 145·6 tons of tetryl were manufactured at H.M. Factory, Queensferry, at the average cost of £297·4 per (short?) ton, the consumption of dimethyl aniline being ·546 (long?) tons per (short?) ton of tetryl produced (*C.T.J.*, 1919, p. 162).

Pentanitromethyl aniline (tetranitromethylphenyl nitramine) has been described (E.P. 3907[10]), but does not seem to have found any application.

Miscellaneous Compounds

The following compounds have been proposed for use as explosives or as ingredients in the manufacture of explosives, and although not in general use they are worthy of short notice with a view to possible future developments:—

Nitromethane, CH_3NO_2.—A stable liquid boiling undecomposed at 101° C. It is readily obtained by the action of sodium nitrite on chloracetic acid (*B.*, **42**, 3438), and owing

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to its low molecular weight would be useful as a means of lowering the freezing point of nitroglycerine. Its high cost precludes its use at present, but experiments carried out by the author indicate that the yields could be greatly improved.

Tetranitromethane $C(NO_2)_4$.—This freezes at 13° C. and boils undecomposed at 126° C. Owing to its large content of oxygen it would be a most valuable ingredient. Several processes have been described for manufacturing it by the action of nitric acid or nitric anhydride on acetic anhydride (D.R.P. 211,198, 211,199, 224,057) or on nitro-aromatic compounds such as picric acid (D.R.P. 184,229), but it is doubtful if any of them are yet technically possible. Tetranitromethane is very toxic.

Nitro-sugars.—These have been studied with a view to their use in detonators. As a class they are soluble in water and hence their isolation is troublesome. They can be purified by crystallization from alcohol, but are too sensitive and unstable for use (*B.*, 1898, 68–90).

Nitro-mannitol.—Mannitol gives a hexanitrate which is insoluble in water and can be crystallized from alcohol. It melts at 112° C. and is comparatively stable. Several attempts have been made to employ it in detonators, but without success so far. It is, however, worthy of further attention.

Nitro-starch.—Many attempts have been made to employ nitro-starch as an ingredient in explosive mixtures, but up to the present no marked success has been obtained. Starch is troublesome to nitrate, as it balls together, and the only satisfactory method is to dissolve it in nitric acid and then spray the solution into sulphuric acid or mixed acid. So far it has been found impossible to stabilize the product on an industrial scale, but as a stable nitro-starch with 14·04 per cent. of nitrogen has been obtained in the laboratory by elaborate treatment with organic solvents it must be concluded that the instability is due to impurities. Nitro-starch is soluble in nitroglycerine, but does not gelatinize it. For further information see *S.S.*, 1910, 82.

Hexanitrodiphenylamine.—This has been used in Germany as a charge for floating mines. It is made by the

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nitration of diphenylamine or by condensing chlordinitrobenzole with aniline and then nitrating the dinitrodiphenylamine thus obtained. It has the objectionable property of being an acid and is extremely poisonous, so that its use is not likely to become general (*S.S.*, 1910, 15; 1913, 205, 231; D.R.P. 86,295).

Hexanitrodiphenyl Sulphide (Picryl sulphide).—This can be obtained by the action of sodium thiosulphate on picryl chloride (E.P. 18,353[13]). It is very stable, non-poisonous, and does not stain yellow. It is decidedly more powerful than picric acid and about 10 per cent. stronger than trinitrotoluol. It has been proposed for use in detonators and as a shell-filling. In connection with the latter, and in view of the use of gas in warfare by Germany, it is interesting to note that the patentee (Carl Hartmann, Schlebusch) claims that "the free sulphurous acid contained in the fumes on the detonation of a striking shell renders the continuance in a closed space, such as casemates, holds of vessels, etc., impossible" (E.P. 18,354[13]). Unfortunately the compound is expensive to produce, as picryl chloride can only be obtained by the nitration of chlorbenzole by using excessive amounts of acid. This might be remedied to some extent by fortifying the waste acid from the trinitration and then using it for mono- or di-nitration. The corresponding *sulphone* is obtained by oxidation with nitric acid (D.R.P. 269,826).

Hexanitrodiphenyl Oxide.—The symmetrical compound (picric anhydride) has not been described, but an unsymmetrical compound is obtained by condensing 1-chlor-2-4-dinitro-benzole with *m*-nitro-phenol and then nitrating (D.R.P. 281,053). It is a stable compound of about the same power as picric acid.

Hexanitro-oxanilide.—This can be obtained by nitrating oxanilide (F.P. 391,106). It is a stable compound melting at 295°–300° C., and is about as powerful as trinitrotoluol. Its temperature of explosion is rather low, and hence it may find useful application as an ingredient of permitted explosives for use in fiery mines.

Hexanitrodiphenyl.—A stable explosive obtained by

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the action of copper powder on picryl chloride. It is too expensive for general use, but may find application for detonators (F.P. 18,333[14]).

Spent Acids.—The manufacture and concentration of nitric and sulphuric acids is treated in another volume in this series,* but although the recovery of spent acids from nitration operations is carried out by similar means it is accompanied by certain difficulties, due chiefly to the organic impurities present. The recovery of spent acids from the manufacture of aliphatic nitric esters is the most simple, as these acids are fairly readily burnt clean. In dealing with nitrocellulose waste acid the strongest portion is revivified for further use by the addition of oleum and nitric acid, and the nitric acid then recovered from the remainder by distillation. The residual sulphuric acid still contains 2–3 per cent. of nitrous acid which cannot be removed by distillation and it is denitrated by steam, the nitrous acid being recovered as nitric acid in towers. The removal of this nitrous acid is absolutely necessary, not only on the grounds of economy, but also to avoid trouble with the lead work of the concentration plant. The denitrated acid is finally concentrated, this being almost invariably done in this country in the Kessler plant, although the Gaillard tower is in use by at least one big firm, and cascade plants are also used.

Nitroglycerine waste acids are not distilled, but are denitrated with steam and then concentrated. Like nitrocellulose waste acids, this is easily accomplished and is usually carried out in the Kessler plant.

Spent acids from aromatic nitrations are much more difficult to deal with owing to the more stable nature of the organic impurities which they contain. In addition the separation of the nitro-compounds is slow and in fact usually continues for several days or even weeks, although the amount that separates is usually comparatively small and hardly worth recovering. The acids usually contain little or no nitric acid, but from 3 to 4 per cent. of nitrous

* Partington, "The Alkali Industry," in this series.

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acid, and hence denitration must be brought about by steam. A certain proportion of the organic matter is volatilized by this means, and as it becomes further nitrated it sets at a comparatively high temperature and causes great trouble in the condensers and towers. This is best remedied by using very capacious condensers and keeping the part of them nearest the towers at about 50°–60° C., but facilities must be provided for cleaning out solid matter, and steam laid on so that the whole plant can be heated up should signs of a stoppage appear. The denitrated acid still contains a large quantity of organic matter, and this tends to separate more readily owing to the greater dilution of the acid. Provision must therefore be made for collecting any that separates, as otherwise it may catch fire in the concentration plant. In any case a considerable amount always distils off during concentration, and hence all fume lines and scrubbers must be run hot to prevent stoppages. To reduce troubles of this nature to a minimum denitration is sometimes carried out with highly superheated steam, about 500° C., and this is undoubtedly the best way, as the high temperature rids the acid more or less of its volatile constituents and not only saves trouble in the concentrating plant, but also makes concentration more easy by reducing the amount of organic matter.

Owing to the stable nature of the organic matter in the sulphuric acid, concentration requires a far higher temperature than is the case with the acids from nitroglycerine and nitrocellulose. For this reason the cascade type of plant is almost invariably employed, and it is usually finished up with a cast-iron pan or pans. These can be fired from the same furnace as the rest of the cascade, but are much better fired separately. The "fume" from these pans is at a high temperature and is sometimes used for denitrating the feed acid.

If the denitration has been carried out at a high temperature so that the denitrated acid is comparatively poor in organic matter, concentration can be carried out in the Kessler plant, but the heavy sludge is very apt to settle out in the plateaux and block up the culottes, so that

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the modified Kessler plants (such as the A.G.D. Concentrator) would probably give better results.

This sludge is chiefly sulphate of iron which has been picked up from the iron nitrators or from the waste acid store tanks when these are made of steel. It is extremely troublesome, as it settles out in various parts of the plant and causes stoppages, and settles out in the basins of the cascade and causes them to crack. It cannot be avoided, but can be reduced somewhat by using lead in preference to iron, especially when handling waste acid. Certainly this results in the formation of lead sulphate, but the amount is much less and it is somewhat less troublesome to deal with.

LITERATURE

Nitroglycerine

A description of some of the older methods of manufacture will be found in "Manufacture of Explosives," O. Guttmann, London, 1905. An account of the modern displacement process will be found in *A.E.*, 1906, p. 90; *J.S.C.I.*, 1908, 193.

Accidents in the manufacture of nitroglycerine have been numerous, and the following Special Reports are very instructive: *S.R.*, 150, 156, 161, 162, 164, 167, 180, 200.

Nitrocellulose

A great deal of information on nitrocellulose for both explosive and other purposes will be found in "The Nitrocellulose Industry," by E. C. Worden, 2 vols., London, 1911.

The displacement process is described in detail in *A.E.*, 1906, p. 77, whereas nitrating centrifuges are described and fully illustrated in *S.S.*, 1910, pp. 352, 413, 434, 458, 478.

The Abel process is described in detail in the *Journal of the Society of Chemical Industry*, 1909, 180. The following Special Reports on accidents will repay study: *S.R.*, 166, 169, 206, 207.

Trinitrotoluol

The following references deal with its stability, use as an explosive, and toxic properties: *A.E.*, 1914, 68; *S.S.*, 1912, 425; 1913, 97, 213; 1914, 239, 378, 405, 432; *P.S.*, xvi., 40, 97, 213.

The majority of accidents that have been due to T.N.T. have taken place during the war and reports are not yet available. The following, however, will be found of interest: *S.S.*, 1907, 333, 413, 416; 1908, 298; 1909, 213; *A.R.*, 1903, 26.

Picric Acid

The best description of the manufacture of picric acid will be found in *Metallurgical and Chemical Engineering*, 1915, p. 686.

For accidents, see *S.R.*, 81, 139.

Tetranitroaniline and Tetryl

A.E., **21**, 21, 36; **24**, 137, 144; **25**, 38.
S.S., 1913, 185.

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Section III.—SMOKELESS PROPELLANTS

Propellants for Rifled Arms.

The properties of a propellant for use with rifled arms are somewhat different from those required by smooth-bore arms, as the former require a slow-burning powder that will impart a "push" to the projectile, whereas shot guns call for a more rapidly burning explosive, the effect of which is more akin to a blow. The projectile in a rifled arm engages closely with the rifling, copper bands ("gas checks" or "driving bands") being provided for this purpose in the case of steel shell, so that comparatively little gas can escape before the projectile leaves the muzzle. If a rapidly burning powder is used combustion will be complete before the projectile has travelled any appreciable distance, and excessive and dangerous pressure will be developed. This pressure, moreover, will fall off rapidly, so that in addition only low muzzle velocity will be attained. If on the other hand a suitable slow-burning powder is employed combustion will take place during the whole travel of the projectile, so that a steady pressure will be maintained and high muzzle velocity without excessive pressure will be achieved. To maintain an even pressure until the moment that the projectile leaves the muzzle is usually mechanically impossible, as it is impossible to construct most guns with the same thickness of metal at the muzzle as at the breach, but the longer the gun the slower burning must be the propellant. In rifled arms the maximum pressure developed is usually about 15 tons per square inch, whereas in shot guns it is only about 2–4 tons.

Before the introduction of smokeless powder, slowness of burning was attained by the use of moulded powders,

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some of the pellets being as much as 1½ in. long. The reduction of surface achieved by moulding the powders caused them to burn much more slowly, as combustion takes place only on the surface. Naturally as the powder burns the pellets become smaller, and consequently the surface less. In order to compensate for this and maintain a steady rate of combustion, they were made with one or more perforations. With these powders, although the external surface decreased during combustion, the internal surface increased, so that by choosing a suitable number of perforations a very steady rate of combustion could be and was obtained. These moulded powders have now gone quite out of use, but the same principle is applied to smokeless powders, viz. the rate of combustion is controlled by the size of the powder.

The following qualities are required of an ideal smokeless powder :—

(1) *Regular Ballistics.*—Regular ballistics are only attained with a regular rate of burning. For this reason the powder must not be so brittle that it breaks up during transport or combustion, and it must not be porous so that combustion can spread through the mass without being confined to the surface. With tubular or perforated powders mechanical strength is necessary, as the gas pressure inside the tubes is greater than that outside, and consequently if the material is mechanically weak the tubes will burst. The effective surface of flake powders may become reduced owing to the flakes lying face to face, and with a view to preventing this some flake powders are made with ridges or knobs. Finally, the powder must contain little or no volatile matter that can evaporate during storage and thereby alter the composition of the explosive, and it should not be affected by climatic conditions, *e.g.* must not be subject to freezing.

(2) *Stability.*—This is very necessary in the case of military powders which may be kept in storage for many years before use. The most stable powders are usually those which are least porus, as porosity frequently leads to instability through oxidation. They are also less hygroscopic.

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(3) *Absence of Fouling.*—The powder must produce a minimum of fouling, and this should be of such a nature that it is easily removed. The products of combustion should contain no substance capable of corroding the barrel.

(4) *Absence of Smoke.*—This can only be achieved by powders which on combustion give only products which are gaseous at the ordinary temperature. All powders produce water on combustion, and in very cold weather this is sometimes condensed to a mist. Any powder containing a nitrate produces more or less smoke owing to solid matter, as ammonium nitrate is the only nitrate giving gaseous decomposition products, and this cannot be used owing to its deliquescent nature.

(5) *Absence of Fume.*—The products of combustion should not contain any poisonous gas. Carbon monoxide is almost always present, but is not dangerous under the conditions under which propellants are used. The muzzle gases of propellants containing nitroglycerine, however, frequently contain a certain amount of this compound, and in consequence give rise to "gun headache." This is particularly the case with heavy ordnance.

(6) *Absence of Erosion.*—Erosion is due to two causes, viz. the scouring action of solid particles and the temperature attained by the interior surface of the arm. The erosion caused by black powder is due almost solely to the scouring effect of the solid products of combustion, whereas in the case of modern smokeless powders erosion is due to the surface of the metal being fused and then washed away by the outrush of the gases, and consequently to avoid erosion the temperature of combustion must be kept down.

(7) *Absence of Back-flash.*—Back-flash is due to the products of combustion being rich in carbon monoxide and this firing when the breach is opened. It is especially dangerous in artillery, and many accidents have been caused by its firing other charges lying in the turret. It can only be avoided by using a powder containing sufficient oxygen to burn all the carbon to carbon dioxide.

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(8) *Absence of Flash.*—Flash is objected to in military explosives, as it reveals the position of the gun when night firing is taking place. It can be remedied by the addition of neutral salts, but these produce smoke which is objectionable by day.

(9) *High Power and Density.*—The greater the power of an explosive, the less is the weight required to produce a given effect. This is important from a military point of view, as it saves transport and means less weight for the soldier to carry. A high density means that less room is required for storage, and is specially desirable for naval purposes where magazine accommodation is limited.

(10) *Insensitiveness to Shock.*—This is also more important for military than for sporting purposes. For use under service conditions a high degree of insensitiveness is very desirable, as the explosive should not explode when struck, for example, by a rifle bullet.

(11) *Quickness.*—The powder should not show any tendency to hang fire when fired with a suitable cap and primer.

Smokeless propellants for rifled arms can be divided into two classes, viz. those consisting of nitroglycerine and nitrocellulose with or without the addition of small quantities of other materials to act as stabilizers or deterrents, and nitrocellulose powders, containing no nitroglycerine, but consisting of nitrocellulose with or without the addition of stabilizers, deterrents, or nitrates. Although both classes contain nitrocellulose, the latter are known as nitrocellulose powders and the former as nitroglycerine powders. The stabilizers are compounds which are capable of taking up the products of decomposition and thus maintaining the powder in a state of neutrality and preventing autocatalysis. For this purpose magnesium carbonate or chalk is sometimes used, especially for stabilizing nitrocellulose, the latter usually being applied by using hard water during the boiling process (p. 50). Of organic compounds, camphor was used at one time, but it is objectionable, as it is volatile, and hence powders made with camphor are liable

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to give irregular ballistics after keeping. It is now almost wholly replaced by diphenylamine or mineral jelly (crude vaseline) although other substances have been proposed, such as diphenylurea, urea, etc.

Both nitroglycerine and nitrocellulose powders consist of colloidal masses of gelatinized nitrocellulose which have been pressed into ribbons, cords, tubes or sheet, these being frequently subsequently cut into flakes when intended for use with small arms. A very lively controversy has arisen as to the relative merits of the two classes of powders, and the question cannot be said to be settled. Against nitroglycerine propellants it has been urged that the fumes of unburnt nitroglycerine cause severe headache, especially in the case of heavy ordnance, that they are liable to freeze at low temperatures and that the high temperature of explosion causes excessive erosion of the gun barrel. In connection with this latter point Vicille carried out quantitative experiments by firing charges in a closed cylinder from which the only escape for the gases was through a hole 1 mm. in diameter in a movable plug. The plug was weighed before and after each shot, and the loss in weight taken as a measure of the erosion. He found that Cordite had a much greater erosive effect than Poudre B (a nitrocellulose propellant), and experiments carried out by Sir Alfred Nobel with special Cordites containing from 10 to 60 per cent. of nitroglycerine showed that within these limits the erosion increased by some 600 per cent. On the other hand, it has been stated that the erosion caused by Cordite M.D., which contains much less nitroglycerine than Cordite Mark I., is only slightly, if any, greater than that caused by the average nitrocellulose powder.

In favour of nitroglycerine powders, it has been urged that they are cheaper to make, give more regular ballistics, and are less liable to produce back-flash than nitrocellulose powders. They keep very well, are not liable to become porous, and the solvent can be almost completely expelled, whereas nitrocellulose powders are liable to become degelatinized and porous, and it is almost impossible to expel the

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solvent by drying for any reasonable time, and hence it is usually necessary to leave from 2–4 per cent. in the explosive. This gradually evaporates on keeping, and its loss naturally alters the ballistics. Nitroglycerine propellants are also more powerful and dense than nitrocellulose powders, so that somewhat less transport and storage space are required for them, great advantages from a military point of view.

It is notable that Great Britain and Italy are the only two first-class Powers that rely on nitroglycerine powders for all classes of arms both military and naval. The other Powers either use nitrocellulose powders exclusively, *e.g.* France, Russia, and the United States, or use nitrocellulose propellants for some arms and nitroglycerine propellants for others, *e.g.* Germany. As the best test of a propellant is its prolonged trial under use, the war should furnish interesting data as to the relative merits of the two classes of powders.

Nitroglycerine Propellants

The first propellant of this class was discovered by Nobel in 1887 and named Ballistite. It was manufactured originally by incorporating collodion cotton with nitroglycerine in the presence of benzole, but the method was soon changed, as it was found that incorporation could be carried out by agitating nitroglycerine with collodion cotton in the presence of hot water, agitation being effected by means of compressed air. The dough thus formed is then rolled between rollers heated at 50°–60° C. until a completely homogeneous and colloidal sheet is obtained, which is finally cut into flakes and usually coated with graphite. The Ballistite thus obtained contains on the average equal weights of nitroglycerine and nitrocellulose, and usually a few tenths of a per cent. of diphenylamine to act as a stabilizer. Owing to the high temperature of explosion Ballistite causes severe erosion, and has now been largely displaced by Cordite and similar explosives containing less nitroglycerine.

Cordite.—Cordite was introduced as the British Service

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propellant in 1889, and originally consisted of nitroglycerine, guncotton, and mineral jelly (crude vaseline) in the following proportions :—

Nitroglycerine	58
Guncotton	37
Mineral jelly	5

In spite of the cooling action of the mineral jelly, however, the erosion caused to the guns was so severe that the composition had to be modified. The Cordite in use at present has the composition—

Nitroglycerine	30
Guncotton	65
Mineral jelly	5

and is known as Cordite M.D. to distinguish it from the original Cordite (Cordite Mark I.), which is still used for some purposes.

The guncotton used in the manufacture of Cordite is usually manufactured from cotton waste by the displacement process and contains 13 per cent. of nitrogen, and 85 per cent. is insoluble in alcohol-ether. The mineral jelly must not flash below 400° Fahr. (204·5° C.) and must not melt completely below 30° C. It must be free from acidity and mineral impurities and its specific gravity at 38° C. should not be less than ·87.

The manufacture of Cordite is carried out in this country as follows, other nitroglycerine propellants being made by very similar methods. The guncotton, after drying and subsequent cooling, is weighed out in the stoves into india-rubber bags holding about 25 lbs. each, lead weights covered with leather or rubber being used. These bags are then carried to the nitroglycerine department, and the required weight of nitroglycerine measured off and poured into each bag. The nitroglycerine can be measured out either in fixed lead measuring vessels or in movable gutta-percha jugs. The bags are then taken to the hand-mixing house, where the contents are emptied out and mixed by hand, being finally rubbed gently through a ½-in. mesh sieve into other bags. These "hand mixings" are much safer than

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nitroglycerine or guncotton alone. and most factories keep a small stock of cordite paste in this form so as to facilitate regular work. The next process consists in incorporating the paste with a mutual solvent so as to gelatinize the nitrocellulose. Acetone has been found most suitable for this purpose and is always used, although at an emergency ether-alcohol could be substituted, and this has been done to a considerable extent during the war. The incorporation is carried out in kneading machines of the Werner-Pfleiderer type (Fig. 15) and takes seven hours. The average charge per machine is 150–250 lbs., and before adding the paste the interior of the machine is moistened with acetone. The paste and the rest of the acetone is then added and the machine set in motion, the mineral jelly being added when the incorporation is half finished, viz. at the end of 3½ hours. The stiff dough heats up somewhat during mixing, but the temperature is not allowed to exceed 40° C. and is controlled by the water jacket with which the mixer is provided. To prevent undue loss of acetone a light wooden cover is placed over the machine. The belts of the machines are liable to become strongly electrified, and ensuing sparks may cause an acetone vapour explosion. To remedy this the machines are earthed and the belts dressed with a mixture of glycerine and water.

When incorporation is complete the charge is placed in bags and removed to the press house, where it is filled into gun-metal cylinders, usually being packed tight by a hydraulic ram, and then pressed through a die in a hydraulic press into cords (hence the name "Cordite") of various sizes or into tubes, a 100-mesh gun-metal gauze being placed above the die to filter out foreign matter. Pressing into tubes is effected by means of a die with a pin in the centre, but when small sizes are being pressed considerable trouble is experienced through a partial vacuum being formed and consequent collapse of the tube. This can be avoided by using a hollow pin with communication to the outer air (E.P. 27,700[10]).

The diameter of the cord depends on the size of the

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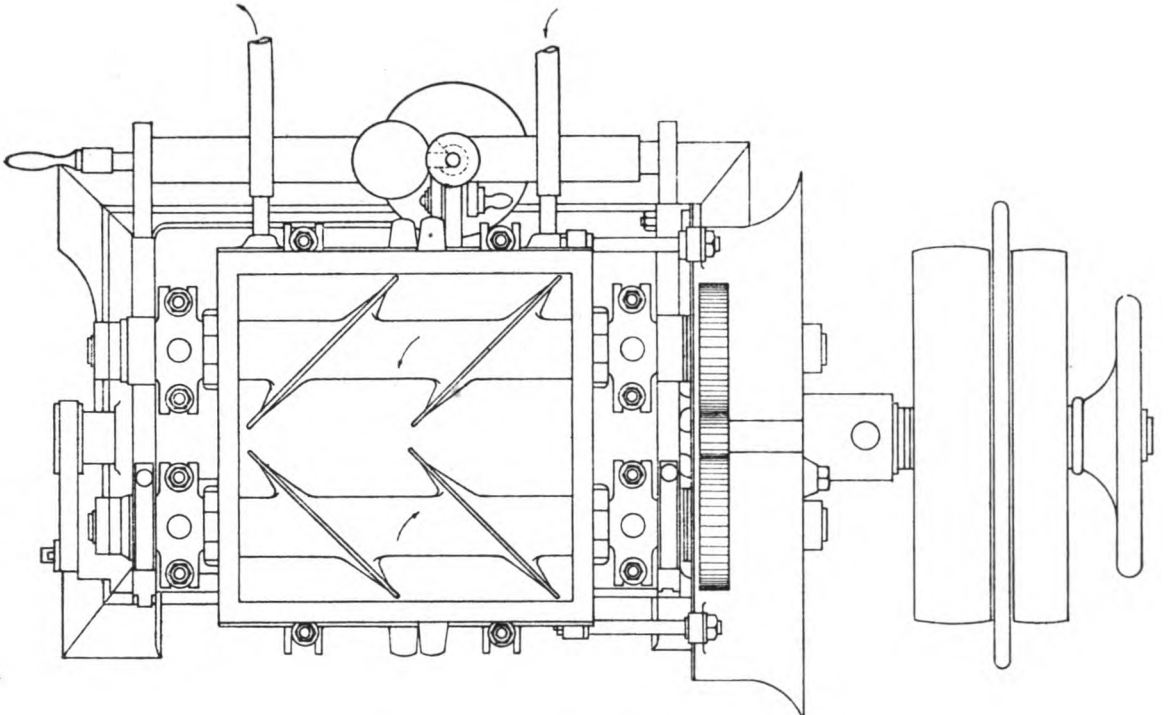

FIG. 15.—Incorporating Machine (Werner and Pfleiderer Type).

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gun for which the Cordite is wanted. The largest size pressed is approximately ·5 of an inch in diameter, but all Cordite is pressed through a die two or three hundredths of an inch larger than the desired diameter of the finished article so as to allow for shrinkage during drying. The smaller sizes of Cordite are reeled on to drums as the cords issue from the press, but the larger sizes are laid out on a table and then cut to the required length. Drying is effected in stoves heated to 40° C. and takes from a few days to two or three months, the time depending on the diameter of the cords, but Cordite Mark I. dries considerably quicker than Cordite M.D.

After drying is complete, different batches of Cordite are blended in order to assure a product with regular ballistic properties. With the smaller sizes which are on reels, such as rifle Cordite, this is very simply done by re-winding several reels simultaneously on to a single drum. The worker carrying out this process should be carefully earthed, as the dry Cordite passing through his hands becomes strongly electrified. The bigger sizes are blended by laying the boxes containing the different batches in a row and then repacking by transferring a handful from each into a different box. The weight per linear inch is usually relied on as a guide for making up blends. Before blending the sticks are carefully picked over by hand, and any which are badly distorted or contain specks of foreign matter thrown out. These specks are chiefly due to brass dust formed by sliding the cylinders over brass guides in the press house, and are difficult to avoid. Such sticks as are rejected in the packing house are re-worked after being incorporated again with acetone.

The various sizes of Cordite used by the British Government are denoted by a fraction, the numerator of which denotes the diameter in hundredths of an inch and the denominator the length of the sticks in inches. For example, size $\frac{45}{25}$ means that the sticks are ·45 in. in diameter and 25 in. long.

Cordite forms a horny mass of brownish-yellow colour. The smaller sizes are flexible, but the larger sizes break

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easily if bent. Cordite Mark I. is much more flexible than Cordite M.D. It is very insensitive to shock, and when not strongly confined burns fiercely, but does not explode. The cost of manufacturing Cordite during the period January —June, 1918, was about £225 per short (?) ton (*C.T.J.*, 1919, p. 189).

In the manufacture of Cordite 56 lbs. of acetone are required for every 100 lbs. of guncotton, and for the sake of economy it is necessary to recover this as far as possible, by suitably treating the air exhausted from the drying stoves. According to one process the acetone-laden air is forced through water contained in a series of closed jars arranged in the form of a cascade, the water and air flowing in contrary directions. By this means the air is robbed of its acetone, and this latter can be recovered by distilling the water with a suitable column. The process in most general use, however, takes advantage of the fact that acetone forms an addition compound with sodium bisulphite, the formula of which is $(CH_3)_2C<^{OH}_{SO_3Na}$. The recovery by this process is carried out by passing the air from the stoves up lead-lined towers which are packed with frames on which strands of wool are woven in a criss-cross pattern (E.P. 25,994[01]). Simultaneously a strong solution of sodium bisulphite is allowed to trickle down the towers, the towers being usually connected in series, and the liquor from the bottom of one pumped to the top of the next. By this means the liquor and air are brought into contact with one another on the counter-current system and very complete scrubbing is effected. After circulating through the towers the liquor is distilled, a little sodium carbonate being first added, and the acetone thus recovered purified by fractionation.

In all recovery processes there are two dangers to be guarded against. One is that fire from one stove may be transmitted to other stoves through the pipe line connecting them with the recovery plant. This can be guarded against by inserting fine wire gauzes in the pipes, these acting on

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the same principle as the Davy safety lamp. It is also advisable to make some sections of the pipe of very flimsy construction, so that they would be broken by an explosion wave. The second danger is that Cordite gives off considerable quantities of nitroglycerine during drying, especially during the later stages, and this is apt to condense and accumulate in the pipes. To avoid this, all pipe lines must be constructed with a fall to one or more points at each of which a catch box is placed from which any accumulation of nitroglycerine can be drawn off safely and conveniently. Some firms make a point of keeping a little paraffin oil of medium consistency (such as medium machinery oil) in these catch boxes, as this has a great effect in moderating the explosive properties of nitroglycerine.

The amount of solvent given off during the later stages of drying large size Cordite, especially Cordite M.D., is so small that it does not pay to recover it. Hence it is usual to transfer these larger sizes to non-recovery stoves after they have lost the greater part of their solvent.

The following gives the name, composition, and physical form of the nitroglycerine propellants used for military purposes by European Powers. The other Powers use nitrocellulose propellants :—

Country.	Propellant.	N.G.	N.C.	M.J.	Form.	Remarks.
Gt. Britain	Cordite Mark I.	58	37	5	Cords, Sticks, Tubes	N.C. has 13 % N.
,,	,, M.D. II.	30	65	5	Cords, Sticks, Tubes	N.C. has 13 % N.
Italy ..	Filite	50	50	—	Cords	1 % Ph_2NH.
,, ..	Solenite	33	64	3		N.C. 12·5 % N.
Germany	Würfelpulver	50	50	—	Cubes	Contains Ph_2NH.
,,	Röhrenpulver	33	64	3	Tubes	

Cordite is very largely used for sporting rifles, and in addition one or two nitroglycerine propellants are manufactured in this country for sporting purposes. Of these *Axite* and *Moddite* are the best known. The former is practically Cordite M.D. to which 2 per cent. of potassium nitrate has been added. The latter contains rather more

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nitroglycerine, and Marshall (" Explosives ") gives the result of an analysis as follows :—

Nitroglycerine	38·7
Nitrocellulose	56·8
Mineral jelly	4·3
Volatile	·2

Nitrocellulose Propellants

These all consist of collodion cotton gelatinized and rendered colloidal by the use of solvents and then pressed into cords, tubes, or ribbons in much the same way as Cordite. Gelatinization can be carried out with acetone, but the colloid thus obtained is very brittle and becomes more so on keeping, so that ether-alcohol is nearly always used. As this does not gelatinize nitrocellulose of high nitrogen content, it is very desirable to use a collodion cotton which is almost completely soluble in ether-alcohol, as otherwise ungelatinized fibres will render the resulting colloid porous. Porosity is usually accompanied by instability as oxidation takes place, and by irregular ballistics and excessive pressures due to combustion spreading through the body of the powder and not remaining confined to the surface.

Nitrocellulose propellants for rifled arms usually contain nothing but nitrocellulose, together with a small amount of stabilizers and a slight percentage of solvent which cannot be removed in any reasonable time in the drying stoves. More rarely they contain a little potassium nitrate, but propellants containing nitrates are more used for shot guns than for rifled arms, and as they are manufactured by somewhat different methods they will be treated separately. As gelatinization is almost invariably brought about by alcohol-ether, the somewhat dangerous process of drying the nitrocellulose can be avoided, the water being displaced by alcohol as described on page 52.

Almost all countries have adopted nitrocellulose propellants for military purposes, and the manufacture of the United States Military Powder serves as a general description of the methods employed.

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U.S. Military Powder.—The nitrocellulose is prepared from cotton waste either by the displacement process or in nitration centrifuges, and contains 12·7 per cent. of nitrogen. It is almost completely soluble in alcohol-ether, and is pulped, washed, and whizzed in the ordinary way. Forty pounds of the wet pulp, which contains 30 per cent. of water, are then packed into a cylinder and treated with alcohol. At first no pressure is applied and the runnings are almost pure water, but after rather more than half a gallon of water has run out pressure at 200 lbs. per square inch, and finally at 3500 lbs. per square inch, is applied. The block or "cheese" thus obtained weighs some 38 lbs. and contains—

Nitrocellulose	73 per cent.
Alcohol	23 ,,
Water	4 ,,

The blocks are roughly broken up by wooden mallets and the disintegration completed by running them in a Werner and Pfleiderer mixing machine (Fig. 15, p. 75) for a quarter of an hour. Each machine takes a charge of three blocks, corresponding to 42 lbs. of pure nitrocellulose. After disintegration is complete 48½ lbs. of ether, in which 6 oz. of diphenylamine has been dissolved, are added to each machine and incorporation carried on for 45 minutes. The charge is then transferred to hydraulic presses and submitted to a pressure of 3500 lbs. per square inch, whereby it is converted into elastic horny blocks weighing about 40 lbs. each, but no solvent is squeezed out during the compression. To get rid of foreign matter, such as wood chips and hard nodules, the colloid is next filtered through a 30-mesh gauze resting on a heavy steel plate perforated with $\frac{1}{16}$-in. holes. Filtration is brought about by applying a pressure of 3500 lbs. per square inch, and the powder issues in the form of cords. These are again pressed into a block at 3500 lbs. per square inch and this block then squirted into multitubular cords in a hydraulic press, the cords having different diameters and a different number of perforations (from 1 to 7), according to the purpose for which the

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powder is required. These multitubular cords are immediately cut up into short lengths by means of revolving knives and dried in stoves.

During the squirting of the cords a great deal of heat is evolved, so that water-cooled dies must be used. It is not advisable, however, to carry this cooling too far, as the cutting knives work best at 30° C.

After squirting and cutting the powder contains about 48 per cent. of solvent, rather more than half of which is ether. Owing to its volatility and to the fact that it does not form addition compounds easily, ether is much more difficult to recover than acetone, and not more than 40 per cent. can be recovered economically, and this only by using condensers served with chilled brine. The temperature of the condensers, however, must not be reduced too far, or the water vapour which comes over simultaneously may be frozen and so cause a stoppage. The ether recovered is mixed with a good deal of alcohol, and is best returned to the ether factory. The finished powder contains from 3 to 7 per cent. of solvent, depending on the size.

Poudre B.—This is the French military powder, and is made from a mixture of soluble and insoluble nitrocellulose gelatinized with ether-alcohol to which some diphenylamine and amyl alcohol have been added. The latter is used as it is left in the finished explosive, and is less volatile than alcohol or ether. After incorporation the mass is worked between rollers at 70° C., and the rolled sheets either cut up in strips or flakes or squirted through a die into ribands. It is then dried at 50° C., and finally frequently washed with water to reduce the amount of solvent.

The powder is decidedly porous, and several disastrous explosions have been attributed to its spontaneous combustion, the most notable of which were the total destruction of two battleships, the *Iéna* in 1907 and the *Liberté* in 1911.

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Shot-gun Smokeless Propellants

Much quicker burning powders are necessary for use with shot guns than with rifled arms, and in order to produce a good pattern, *i.e.* in order that the shot may not spread unduly, comparatively little pressure must be present at the moment that the charge leaves the muzzle.

Almost all shot-gun smokeless propellants are nitrocellulose powders to which as a rule a certain amount of barium or potassium nitrate has been added, and they may be roughly divided into two classes, viz. condensed powders and bulk powders. The former are completely gelatinized powders and are made by methods very similar to those used for rifle propellants, the necessary increased rate of combustion being attained by forming them into thin flakes or small grains. Munroe describes the manufacture as being carried out in the United States as follows :—

Finely pulped wet nitrocellulose is mechanically agitated with water containing 5 per cent. of barium nitrate and 2 per cent. of potassium nitrate in a vertical jacketed pan. An emulsion of amyl acetate and water containing barium and potassium nitrate is then added, and granulation allowed to take place at the ordinary temperature for a few minutes, after which steam is turned into the jacket and agitation and heating continued for 5–6 hours, during which time most of the amyl acetate and some water distil off and are condensed. The contents of the vessel are then run out and the grains dried and sieved, oversized and undersized grains being added to the next charge in the proportion of 250 lbs. of waste to 450 lbs. of fresh nitrocellulose.

Another American sporting powder, Indurite, is made from guncotton, the soluble portion being first extracted with methyl alcohol and the residue then gelatinized with nitrobenzole, this latter being finally removed by treatment with hot water.

One of the great disadvantages of condensed powders lies in the fact that owing to their small bulk they require special cartridge cases. Also in loading cartridges the

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powder is always measured out, and consequently the higher the density of the powder the greater is the error in weight due to slight differences in volume. For this reason "bulk" powders are usually preferred to the condensed powders. These are composed of grains of nitrocellulose, with or without the addition of small quantities of other substances, the grains being gelatinized on the surface. Such bulk powders are known as 42-grain and 33-grain powders, meaning that the charge for a 12-bore gun is 42 and 33 grains respectively, and that the volume occupied by this amount is the same as that of the standard black powder charge, viz. 3 drams. Thirty-grain powders are also made, but in these gelatinization is almost complete, so that they are more in the nature of condensed powders.

Most shot-gun powders contain barium and/or potassium nitrate, and in many cases it is found best to incorporate a much greater quantity than is desired in the finished powder, the excess being subsequently removed by dissolving out with water. This yields a more bulky powder, and facilitates the production of one having a correct weight for a given volume.

The nitrocellulose used for shot-gun powders usually contains from 12·0 to 12·8 per cent. of nitrogen, but the nitrogen content of nitrolignite powders is frequently lower than this.

The first process in manufacturing bulk powders is graining the nitrocellulose. This can be done in several ways, such as grinding in edge runners while wet or in ball mills, the balls being made of lignum vitæ. During this process the other ingredients, such as nitrates, are added and the incorporating continued until mixing is complete and the grains are judged to be suitable for the class of powder desired. They are then roughly sifted and dried in stoves at a low temperature. After drying they are again sifted to remove dust and then gelatinized on the surface. This is effected in revolving drums provided with ridges so as to keep the powder in motion. The solvent is generally sprayed in, as if added in bulk it does not become evenly distributed, with the result that some of the grains

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remain ungelatinized, while others become overgelatinized and may adhere together and form lumps. After spraying and thorough mixing so as to ensure even distribution of the solvent, the grains are allowed to steep for a few hours and then dried. The drying is best carried out in two stages, as during the later stages the amount of solvent given off is so small that it is not worth recovering. In order to recover as much solvent as possible, the first part of the drying is carried out in revolving drums *in vacuo*, the vacuum pump being connected with the hollow spindle of the drum through a copper coil condenser cooled with chilled brine. A fine gauze is interposed between the drum and the condenser in order to prevent dust being carried over mechanically. In order to guard against pressure being set up in the drum through the condenser becoming frozen or the dust filter choked, the cover of the charging hole is held in position by the vacuum only, so that should the vacuum fail it falls off and at once releases any pressure. After as much as possible of the solvent has been recovered, the powder is removed to stoves and the drying completed by spreading it in thin layers on shallow trays, no attempt being made to recover solvent, as it is given off too slowly. When drying is complete the powder is again sifted to remove oversized and undersized grains and then allowed to age for about six weeks. The ageing period is necessary in order that the powder may take up a certain amount of moisture from the atmosphere, and reach the state in which it will be used. The ballistic properties of each batch are then tested, and the various batches carefully blended so as to produce a powder with standard properties. This blending is most essential if different lots of the same powder are to produce the same effect in use. The production of 30-grain powders involves the use of nitrocellulose with a high nitrogen content in order that the smaller weight of powder may produce the same power, and to reduce the rate of burning gelatinization must be complete. At the same time the bulk of the powder must be maintained in order that a 30-grain charge may occupy the standard volume of 3 drams.

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This is effected by incorporating the nitrocellulose with several times its weight of barium or potassium nitrate, and then drying and gelatinizing freely. The dough is then pressed in much the same way as a condensed powder, and after drying the nitrate is almost completely removed by treatment with warm water. The same method is used in making 33-grain powders, although the gelatinization is not so complete.

As regards the solvents used in preparing bulk powders, acetone alone is not very suitable, as it produces a brittle colloid. It is, however, used sometimes in conjunction with alcohol. Alcohol-ether mixture gives excellent results, but is very difficult to recover. Ethyl acetate and amyl acetate are both used to a large extent either alone or in conjunction with other solvents, and the use of benzole has also been proposed. Sometimes an aromatic nitrohydrocarbon, such as dinitrobenzole or dinitrotoluol or trinitrotoluol, is added to the solvent in order to assist the gelatinization. Such compounds naturally remain in the powder, and consequently when used sufficient nitrate for their combustion must be added. A certain amount of an organic dyestuff, such as aurine, is also generally added to the solvent in order to improve the appearance of the powder. In some cases also a solution of collodion is added, so as to varnish the outsides of the grains. This makes them somewhat less liable to take up moisture, and at the same time slows down the rate of combustion. This latter is also effected by glazing the finished powder by rumbling with a little graphite. The table on page 86 shows the composition of a few well-known sporting powders manufactured in this country.

Of the 33-grain powders, Smokeless Diamond is probably the most used in this country. It chiefly consists of nitrocellulose and barium nitrate, the grain being glazed with graphite.

Schultze Powder, which was one of the earliest smokeless shot-gun propellants, is a nitro-lignine powder and is made from wood. The wood is first carefully purified to free it as far as possible from pectose and non-cellulosic substances

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and then nitrated. After washing the nitro-lignine is impregnated with barium nitrate, and the grains then gelatinized on the surface and hardened by spraying with ethyl-alcohol or other suitable solvent. Schultze Powder has always been a favourite with sportsmen for use with the shot gun, but up to the present nitro-lignine has not been found suitable for use with rifled arms. Probably the German Government has evolved some method of rendering it suitable, as the Allied blockade cut off the supply of cotton.

	Imperial Schultze.	Amberite.	E.C.	Schultze.	Kynoch's Smokeless.	Sporting Ballistite.
N.G.	—	—	—	—	—	37·6
N.C.	—	71	79	62·1	52·1	62·3
Nitro-lignine ..	80·1	—	—	—	—	—
D.N.T.	—	—	—	—	19·5	—
KNO_3	—	1·2	4·5	1·8	1·4	—
$Ba(NO_3)_2$	10·2	18·6	7·5	26·1	22·2	—
Camphor	—	—	4·1	—	—	—
W.M.	—	1·4	3·8	—	2·7	—
M.J.	7·9	5·8	—	4·9	—	—
Starch	—	—	—	3·5	—	—
Carbon	—	—	—	—	—	—
Ash	—	—	—	—	·9	—
Volatile	1·8	2	1·1	1·6	1·2	—

Schultze cube Powder is a 30-grain fully gelatinized powder, and is probably derived from cotton or wood pulp. In France, where the manufacture of explosives is a State monopoly, the following sporting powders are provided :—

	Poudre S.	Poudre J.	Poudre M.	Poudre T.
N.C.	65	83	71	100
$Ba(NO_3)_2$	29	—	20	—
KNO_3	6	—	5	—
$Am_2Cr_2O_7$	—	14	—	—
$K_2Cr_2O_7$	—	3	—	—
Camphor	—	—	3	—
Gélose	—	—	1	—
Number of grains per gram ..	1000	250–300	3500	2500
Charge for 16-calibre gun with 30 grams No. 6 shot ..	2·4	2·8	2·1	1·9
Density	·500	·750	·475	·565
Mean pressure, Kg. per cm.^{2}..	445	350	500	470

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Poudre T bis is very similar to Poudre T, but is rather quicker.

Poudre S is made by mixing the ingredients wet under light edge runners and then drying and incorporating with ethyl-alcohol, the dough being formed into grains by passing it through a sieve. It produces a great deal of fouling.

Poudre J is made by incorporating the nitrocellulose with ether-alcohol and very finely ground bichromates and then rolling out.

Poudre M is made in much the same way as Poudre S, but the grains receive a final treatment with ether-alcohol in which camphor and collodion are dissolved. It is a powder which is apt to develop excessive pressures.

Poudre T contains no nitrate or bichromate, and the gelatinization is brought about by a mixture of ethyl acetate and acetone.

Of the Belgian sporting powders, Mullerite, which contains no mineral salts, and Clermonite, which is a mixture of nitrocellulose and barium and potassium nitrate, are the best known.

Several German powders have acquired a good reputation. Among them may be mentioned Fasan and Tiger, which are roughly 42-grain powders; Rothweil and Walsrode, 33-grain powders; and Saxonia and Adler-Marke, 30-grain powders. They are made in much the same way as the corresponding British powders. Walsrode is gelatinized with ethyl acetate.

Aliphatic Solvents

The solvents used in the manufacture of smokeless propellants are expensive and add considerably to the cost of manufacture. *Alcohol* up to the present has been obtained by the fermentation of sugars or starches, molasses, potato and maize being the chief raw materials. Recently, however, sawdust and the sulphite liquor obtained as a waste product in the manufacture of paper pulp has been used, as on hydrolysis with acids the cellulosic substances are broken down into fermentable sugars. Considerable difficulties have been met with in carrying out the process on a commercial

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scale, but these are gradually being overcome, and a good deal of attention is being paid to the subject in the United States. A purely synthetic process is also in course of development, and consists in converting acetylene into acetaldehyde and then reducing this to alcohol.

Ether has usually been manufactured by the action of sulphuric acid on alcohol, but probably the catalytic dehydration of alcohol will prove to be a less costly process in the future.

Acetic acid, used for the manufacture of ethyl acetate and acetone, is usually obtained as a by-product in the carbonization of wood. Unfortunately coniferous woods are quite unsuited for the purpose, and even with hard woods the yield is very poor. Here again attempts are being made with considerable success to introduce purely synthetic methods. In these the starting out substance is acetylene, which, when treated with sulphuric acid in the presence of mercuric sulphate, takes up water and passes into acetaldehyde, this being readily oxidized to acetic acid—

$$\begin{matrix} CH \\ \| \\ CH \end{matrix} \rightarrow \begin{matrix} CH_3 \\ | \\ CHO \end{matrix} \rightarrow \begin{matrix} CH_3 \\ | \\ COOH \end{matrix}$$

The process is an old one, but it is only during the last two or three years that any success has been achieved, and it is still too early to predict to what extent the process is likely to come into general use. One of the great troubles is the tendency of the aldehyde to polymerize the resinous substances.

Acetone has generally been manufactured by the dry distillation of calcium acetate, this decomposing at 300° C. into the carbonate and acetone—

$$(CH_3CO)_2Ca = CaCO_3 + (CH_3)_2CO$$

Acetone, accompanied by fusel oil, can, however, also be obtained by the fermentation of starch, and seaweed and horse-chestnuts also appear suitable for the purpose. In Great Britain a company was floated in 1912 to work the process, the primary object, however, being to obtain the

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fusel oil and from it synthetic rubber. This latter part of the scheme does not seem to have been a success, but the company has produced acetone for war purposes. In the United States a large plant for the fermentation of seaweed has been put down by the Herculose Powder Company on the Pacific sea-board, potash being obtained amongst other things as a by-product, and in Great Britain experimental work has been done at Penzance in Cornwall. In these processes the residues, after the extraction of the valuable products, are burnt in gas producers and so furnish a large percentage of the heat required for running the works. Iodine and bromine are also recovered, but for this purpose the weed should be cut from the sea bottom at a considerable depth, as that washed up on to the shore is comparatively poor in these elements.

LITERATURE

The manufacture of Cordite is described in detail in "Treatise on Service Explosives," 1907, published by Authority, and an illustrated description of the New Explosives Company's Cordite plant appeared in *Arms and Explosives*, 1898, p. 242.

Litigation on the Cordite patents is referred to in *A.E.*, 1897, pp. 89, 106, 116, 172.

The manufacture of the United States Military Smokeless Powder is given in detail by E. C. Worden in "Nitrocellulose Industry," vol. ii., 1911; and also in the *Journal of the United States Artillery*, 1910, p. 140.

The use of nitromethane for assisting gelatinization is covered by F.P. 394,992. The addition of nitroguanidine for reducing the temperature of explosion is covered by U.S.P. 899,855, and the use of tartrates for the same purpose by E.P. $15{,}565^{05}$.

Producing flaked powders with knobs or ribs to prevent the flakes lying face to face is the subject of E.P. $12{,}892^{05}$, $21{,}779^{05}$.

Experiments on the stability, or lack of stability, of Poudre B are described in *P.S.*, xv., 1; *S.S.*, 1910, pp. 345, 369, 389; 1911, pp. 303, 327, 441, 464.

Vielle's experiments on erosion are described in *P.S.*, xi. 157.

Smokeless propellants in general are discussed in *S.S.*, 1913, pp. 126, 285, 307, 330, 352, and the relative merits of nitroglycerine and nitrocellulose propellants in *S.S.*, 1913, pp. 368, 393.

Experiments on the behaviour of smokeless propellants when burnt in quantities of from 1 to 10 tons are described with photographs in *S.S.*, 1914, pp. 187, 217.

A large number of works on interior and exterior ballistics, most of them more or less mathematical, have been published from time to time, but the following can be recommended:—

J. M. Ingalls, "Interior Ballistics," New York, 1912.

C. Cranz, "Lehrbuch der Ballistik," 3 vols., Berlin, 1910–1913.

P. Charbonniere, "Balistique Intérieure," Paris, 1908.

A. C. Crehore and G. O. Squier, "Polarizing Photo-Chronograph," New York, 1897.

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Section IV.—BLASTING EXPLOSIVES

For blasting hard rock a powerful and brisant explosive is required, more especially in operations such as tunnelling, where it is not required to obtain the material in large pieces, and an explosive with high density is also desirable for this class of work, as the cost of making bore holes in hard material is very considerable. Softer minerals require a slower burning explosive, as otherwise they are shattered too much and excessive amounts of dust are produced. For this reason and on account of its cheapness, gunpowder is still extensively used for quarrying. For work in coal mines a mild explosive is required for coal getting in order that the material shall not be unduly shattered, and a more brisant explosive for ripping and clearing away stone. At the same time the explosive used must not fire the mine gases nor cause a coal-dust explosion. Explosives for use in fiery mines are known in this country as Permitted Explosives, and are treated in the next section.

For use in wet situations a satisfactory blasting explosive should not be readily spoilt by water, although this trouble can be largely got over by using waterproof wrappers. Finally, as blasting explosives are largely used by ignorant people, they should be as fool-proof as possible. Explosives containing nitroglycerine are liable to freeze, and in this state are difficult to detonate. There is a statutory obligation to thaw out such explosives before use in a proper warming pan, but in spite of this accidents occur every year through miners thawing out frozen explosives over a fire. The warming pans are very simple in construction and consist of felt-covered, double-walled tin-plate vessels, the explosive being placed in the inner vessel and warm water in the annular space. By their use explosives can

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be thawed in safety, but the process takes time, and to some minds the temptation to thaw over a fire seems irresistible.

Except in the case of very heavy charges, the danger from fumes is not great when surface blasting is carried on, but in underground workings the case is different. The poisonous fumes that result from the detonation of an explosive include carbon monoxide, due in part to lack of oxygen and in part to the paper wrapper. Lack of oxygen in the explosive mixture can be remedied by the addition of more oxidizing agent, but it is not safe to remove the wrapper before inserting the cartridge into the bore hole. Metal foil wrappers might be substituted for paper, but this would only be changing one evil for another, as the metallic oxide in itself would be objectionable unless aluminium or tinfoil were used. The only satisfactory safeguard would seem to lie in ample ventilation. Explosives containing barium or lead salts give off poisonous fumes due to the salts of these metals, and ammonium perchlorate explosives are very liable to evolve chlorine unless alkali nitrate is also present in sufficient quantity to provide a base for the chlorine to unite with. Finally, explosives containing nitro-aromatic hydrocarbons may give off poisonous fumes of these if detonation is incomplete through insufficient oxygen or through the use of too weak a detonator.

All composite blasting explosives consist of an oxidizing agent and oxidizable matter, any or none of which may be explosives in themselves. In gunpowder, for example, the sulphur and charcoal provide the combustible matter and the nitre the oxidizing agent, none of these being capable of explosion alone. Blasting Gelatine, on the other hand, consists of nitrocellulose as combustible matter and nitro-glycerine as an oxidizing agent, both these ingredients being capable of exploding alone. An intermediate stage is represented by Gelignite, in which an explodable and a non-explodable oxidizing agent (nitroglycerine and potassium nitrate) is used in conjunction with explodable and non-explodable combustible matter (nitrocellulose and wood meal).

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The following list gives the chief oxidizing agents available industrially, together with the maximum amount of available oxygen per 100 grams :—

N.G.	.. 3·5	$KClO_3$	 37
KNO_3	.. 40	$NaClO_3$	 45
$NaNO_3$	.. 47	$KClO_4$	 46
$AmNO_3$	.. 20	$NaClO_4$	 52
$Ba(NO_3)_2$	.. 30	$AmClO_4$	.. 27
$Pb(NO_3)_2$	.. 24 (PbO)	$Ba(ClO_4)_2$	.. 38
$Pb(NO_3)_2$	.. 29 (Pb)		

Of these it should be noted that nitroglycerine, ammonium nitrate, and ammonium perchlorate are all capable of being exploded, although ammonium nitrate is only exploded with difficulty. The chlorates are also capable of exploding, as they are endothermic compounds and one or two accidents have been due to chlorate explosions.

A large number of combustible matters is available, of which the following are the most important :—

Nitrocellulose.
Dinitrobenzole.
Di- and tri-nitrotoluol.
Mono- di- and tri-nitronaphthalene.
Wood meal.
Charcoal.

To a lesser extent starch, flour, potato meal, and the like are also used, as they help the resulting mixture to bind together. Castor oil enters into the composition of certain chlorate mixtures, as it renders them less sensitive to shock.

Finally, inert substances such as kiesselguhr, silica, mica, etc., are used for absorbing nitroglycerine in some explosives such as Dynamite, and neutral salts such as sodium chloride are used as "coolers" in Permitted Explosives.

Blasting explosives, other than black powder, can be roughly divided into four classes, viz. : (1) Dynamite and its Congeners ; (2) Gelatinized Explosives ; (3) Chlorate Mixtures ; (4) Ammonium Nitrate Explosives. The division is not a very sharp one, but is the most convenient method of classification.

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Dynamite and its Congeners

When Nobel first introduced nitroglycerine as a blasting explosive it was used in the liquid form, but was frequently transported in a frozen condition in order to minimize the danger. In spite of all precautions, however, numerous accidents occurred, so that the transport of nitroglycerine either in the liquid or in the frozen state was soon forbidden. This caused Nobel to seek some inactive substance that would absorb the oil and render its conveyance and use safe without interfering with its explosive properties. He found kiesselguhr, a form of siliceous earth found in Germany, Austria, Norway, Australia, and Scotland, to be most suitable, as good qualities will take up three times their weight of nitroglycerine and still remain dry; but in his patent he also claimed the use of other materials such as ground brick, dry plaster, etc. He named the explosive thus obtained Dynamite.

Kiesselguhr, or guhr as it is usually called, varies very much in quality, some samples being quite unsuited for Dynamite manufacture owing to their very poor absorptive power or high content of sand. The content of organic matter is also very variable, running from 4–5 per cent. to as high as 35 or 40 per cent. in the case of many of the Scotch deposits. In any case, the guhr requires to be thoroughly calcined before use in order to burn this off, and must then be sifted through a 30-mesh sieve in order to get rid of sand. This latter process is absolutely essential, as grains of sand would give rise to undue friction when making up the cartridges and might well cause an explosion. After calcining the guhr usually has a pink colour due to oxide of iron, but some samples are almost white, and when this is the case most manufacturers add a little red ochre in order that the finished Dynamite may have the characteristic red-brown colour.

The manufacture of Dynamite is carried out as follows: The guhr is weighed out after sieving into wooden boxes or rubber bags and taken to the nitroglycerine department,

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where the requisite quantity of nitroglycerine is measured out and added. The boxes or bags are then carried to the mixing house, where they are emptied out into lead tanks and the contents thoroughly mixed. This can be done by hand by rubbing the loose Dynamite repeatedly through a coarse sieve (about 8-mesh), or after a rough mixing by hand the loose Dynamite can be transferred to a Werner and Pfleiderer mixing machine (Fig. 15, p. 75) and mixing then completed mechanically. The resulting powder should not feel moist, but at the same time it must not be too dry or it will not bind properly in the dynamite pumps and will not detonate easily in use. If too moist more guhr can be added or from ·5 to 1 per cent. of barium sulphate can be mixed in, as this lowers the absorptive capacity of the guhr. If too dry more nitroglycerine must be added. With a little experience it is easy to judge whether the explosive is of the right consistency or not. Dynamite mixing is an extremely unpleasant occupation for those not used to it, as the dusty explosive enters the mouth and nose and causes very severe headache. Those engaged continuously in the process as a rule get acclimatized, but when, as is often the case, the explosive is only manufactured intermittently the workers suffer severely, even those who have been used to handling nitroglycerine jellies getting headache at first when put on to Dynamite.

For converting the loose Dynamite into cartridges the Guttmann dynamite pump (Fig. 16) is almost universally used. It consists of a plunger working vertically between two loose guides, motion being imparted by a horizontal lever. The end of the plunger is shod with lignum vitæ and works in a brass tube of the same diameter as the finished cartridge, plenty of clearance being allowed between the plunger and the brass tube so as to avoid friction. The upper end of the brass tube is fixed into a brass block, usually covered with leather, to which is attached the lower end of a conical bag made of cloth or light leather. The upper and wider end of this bag is attached to a boss further up the plunger by means of three strings, so that at each

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upward stroke of the plunger the bag gets a jerk. An inverted bell is fixed to the plunger just above the bag, so as to prevent explosive working up into the guides.

In pumping cartridges the loose Dynamite is fed into the bag with a wooden scoop, and the plunger then worked up and down by hand. Each upward stroke jerks some of the powder into the brass tube, which the following down-

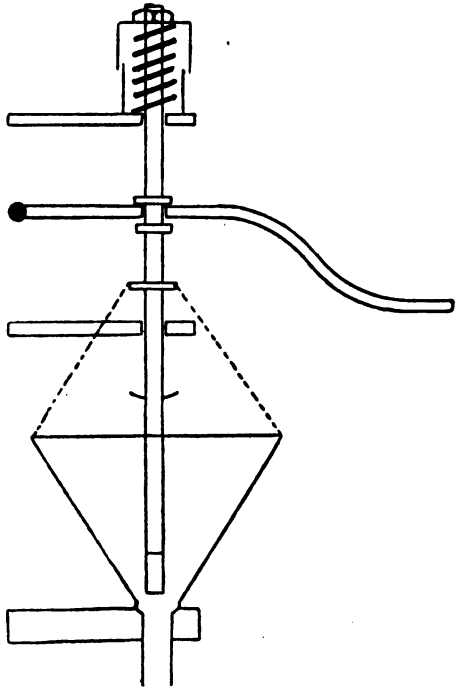

FIG. 16.—Dynamite Pump.

ward stroke compresses into a more or less coherent mass which issues at the lower end as a rod. This is broken off from time to time and wrapped in parchment paper. The most usual diameters for Dynamite cartridges are $\frac{3}{4}$ in. and $\frac{7}{8}$ in., and the most usual weight 2 oz. Ordinary Dynamite, consisting of 75 per cent. of nitroglycerine and 25 per cent. of guhr, with the possible addition of small quantities of

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ochre for colouring, or a trace of calcium or magnesium carbonate to render it more stable, is known in Europe as Dynamite No. 1 to distinguish it from the less powerful Dynamites formerly made to some extent, viz. Dynamite No. 2, consisting of 18 per cent. of nitroglycerine, 10 per cent. of carbon, and 72 per cent. of nitre; and Dynamite No. 3, consisting of 37·5 per cent. of nitroglycerine, 12·5 per cent. of guhr, and 25 per cent. each of wood meal and nitre. In the United States, however, guhr Dynamite goes by the name of Giant Powder, the name Dynamite being reserved for mixtures of nitroglycerine, wood meal, and sodium nitrate.

In France, in addition to guhr Dynamite, Dynamites are also made from a mineral called randanite, composed of weathered felspar, with or without the addition of absorbent silica either from natural sources or manufactured by passing silicon fluoride into water. The Government factory at Vonges, for example, makes the following grades:—

	No. 1.	No. 2.	No. 3.	Special.
N.G.	75	50	30	90
Randanite	20·8	—	—	1
Silica	3·8	48	65	8
$MgCO_3$	·4	—	—	1
$CaCO_3$	—	1·5	1	—
Ochre	—	·5	5	—
Slag	—	—	4	—

In Spain mica is sometimes used as an absorbent, the product containing 42 per cent. of nitroglycerine.

Dynamite had a great vogue when first introduced, but is comparatively little used now as it has been largely replaced by gelatinized explosives, although there is still a fair demand for it. It freezes more easily than nitroglycerine, and in the frozen state is difficult to detonate. It is very brisant in nature, and has the great disadvantage that in contact with water the nitroglycerine is displaced.

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In order to utilize the excess of oxygen present in nitroglycerine many powders have been made in which a combustible absorbent has been used in place of guhr. Charcoal made from cork has great absorptive power, and an explosive consisting of 90 per cent. of nitroglycerine and 10 per cent. of cork charcoal was manufactured at one time under the name of Carbo-dynamite. It was too violent and expensive for most purposes, however, and wood meal is more usually employed. Atlas Powder, for example, consists of nitroglycerine 75 and wood meal 21 per cent., the balance being made up of chalk with or without the addition of a little sodium nitrate.

These wood meal Dynamites are deficient in oxygen, and in order to supply this deficiency nitrates are added. This class of explosive has become extremely popular in the United States, where many blasting explosives are made consisting of varying quantities of nitroglycerine, wood meal, and sodium or ammonium nitrate. Sodium nitrate is preferred to the potassium salt on account of its lower price, but it is decidedly hygroscopic, and ammonium nitrate is still more so, so that these explosives readily take up water which in turn displaces the nitroglycerine. For this reason they have never come into use in this country.

The following table gives a few examples of American Straight Dynamites, but it must be borne in mind that the figures are only average ones and considerable divergence is often met with :—

Dynamite.	70 per cent.	60 per cent.	50 per cent.	40 per cent.	30 per cent.
N.G.	70	60	50	40	30
W.M. ..	20	16·5	14	12	10
$NaNO_3$ ..	7	22·5	35	47	59
Na_2CO_3 ..					
$MgCO_3$ ..	3	1	1	1	1
$CaCo_3$ ..					

Of these 40 per cent. Dynamite is probably the most used, and is frequently known as Hercules Powder.

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Other well-known American blasting powders of this class are—

	Carbonite.	Safety Nitro.	Stonite.
N.G.	25	70	68
W.M.	40·5	12·6	4
$NaNO_3$	34	17·4	—
KNO_3	—	—	8
Guhr	—	—	20
Carbonate	·5	—	—

An explosive of this class much used in South Africa is Ligdyn, which has the composition—

N.G.	40
W.M.	13
$NaNO_3$	45
Wheat flour	2

As will be seen, it is very similar in composition to the American 40 per cent. Dynamite or Hercules Powder. Similar mixtures are also used in the United States in which about half the nitroglycerine has been replaced by ammonium nitrate. These are known as Ammonia Dynamites, and are made in several grades, 40 per cent. Ammonia Dynamite, for example, containing about 20 per cent. of nitroglycerine and the same amount of ammonium nitrate, the balance being made up of sodium nitrate and wood meal.

Another favourite type of explosive used in America is a kind of crude gunpowder to which nitroglycerine has been added. The gunpowder base is composed of sulphur and sodium nitrate, and powdered coal is frequently substituted for charcoal. In some cases wood meal is added to assist the absorption of the nitroglycerine. The following are typical examples of this class of explosive :—

	Judson Powder.	Vulcan Powder.	Stump Powder.	Low Powder.
N.G.	5	30	20	5
$NaNO_3$	64	52·5	50	70
S	16	7	5	7
Charcoal	—	10	—	—
Coal	15	—	20	18
W.M.	—	—	5	—

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Gelatinized Explosives

In 1875 Nobel discovered that collodion cotton was capable of dissolving in nitroglycerine to form a jelly. This discovery, which was of an accidental nature and due to Nobel having cut his finger and closed the wound with collodion prior to his carrying out some experiments with nitroglycerine, has had a very far-reaching effect on the explosives industry, as it must be regarded not only as the forerunner of all gelatinized blasting explosives, but also of the nitroglycerine propellants.

Gelatinized explosives have several great advantages over Dynamite. In the first place they dispense with all inert ingredients, so that the whole of the explosive is of an active nature. Hence much more powerful explosives can be obtained, but at the same time these are easier to dope down by the addition of wood meal and nitrates. The jellies are far less sensitive to moisture than Dynamite and can remain in contact with water for a reasonable time without the nitroglycerine being displaced, and at the same time they are less sensitive to shock. The labour involved in making them up into cartridges is much less than is the case with Dynamite and is also much safer, as the somewhat dangerous dynamite pump is not required.

Up to the present all gelatinized explosives have contained nitroglycerine, but jellies can also be obtained by warming collodion cotton with certain nitroaromatic hydrocarbons such as the so-called liquid trinitrotoluol. Unfortunately these jellies are very difficult to detonate and are not very stiff, and so have not as yet found any application. Future research, however, may enable these disadvantages to be overcome and thus render possible a gelatinous explosive containing no nitroglycerine.

In manufacturing gelatinous explosives the dry collodion cotton is weighed out into bags and carried to the nitroglycerine department, where the requisite quantity of nitroglycerine is measured out, poured on to the collodion,

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and the whole mixed by hand. The thin liquid is then carried in gutta-percha buckets with light wooden lids to the mixing house, where it is transferred to water-jacketed lead tanks. The water in the jacket is kept at about 50° C., but the temperature of the explosive should not exceed 40°–45° C. Gelatinization is allowed to proceed for some hours, usually overnight, and the charge then transferred to a mixing machine and mixed mechanically for an hour, any dope, such as wood meal and potassium nitrate, being added at this point. The machines are best jacketed with warm water to prevent the jelly becoming too stiff, and are usually of the Werner and Pfleiderer type shown in Fig. 15,

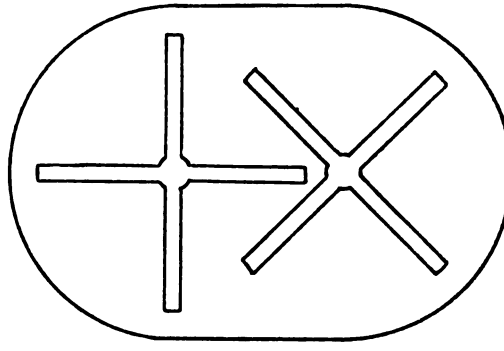

FIG. 17.—McRoberts Type Incorporating Machine (Plan).

p. 75. To avoid all chance of nitroglycerine entering the bearings, however, the McRoberts mixer, shown diagrammatically in Figs. 17 and 18, is sometimes preferred. In this case the explosive mixing pan is carried on a platform that can be lowered so that the mixing arms clear the top of the pan and allow it to be withdrawn. In charging the machine the pan is withdrawn and the jelly loaded into it, after which it is wheeled into place and the platform raised. Stops are provided to prevent the pan being raised too high and consequently the revolving arms coming in contact with the bottom. Opinions differ as regards the merits of the two types of machine, but the Werner and Pfleiderer type is usually preferred and would seem to cause no more

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accidents than the McRoberts type. They are specially built for the purpose, and precautions are taken to guard against explosive entering the bearings. The McRoberts machines have the advantage that the whole process of gelatinization and incorporation can be carried out in the same tank, thus avoiding transferring the jelly from the gelatinization tank to the incorporating machine; but, on the other hand, having to move a heavy article like the mixing

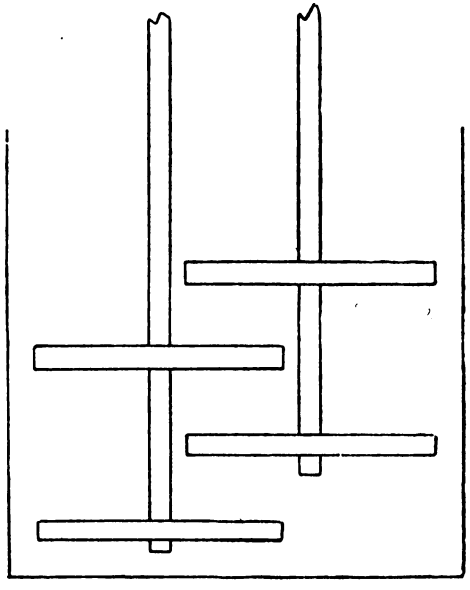

FIG. 18.—McRoberts Type Incorporating Machine (Sectional Elevation).

tank is decidedly objectionable. When incorporation is complete the jelly is transferred to wooden boxes and carried to the cartridge huts, where it is put through "sausage" machines and the resulting cord cut up with double-bladed brass knives and then wrapped in parchment paper.

These "sausage" machines (Figs. 19 and 20) are constructed of gun-metal in such a way that all bearings are external so that no explosive can get into them, and sufficient clearance is left where the shaft enters to avoid friction.

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They are provided with internal ribs to prevent the jelly from merely being revolved, and are always worked by hand. The conical type (Fig. 19) was the original type used, but the circular type (Fig. 20) is now generally preferred, as it puts less pressure on the explosive. In making the usual size of cartridge, viz. $\frac{3}{4}$ in. or $\frac{7}{8}$ in., two nozzles are used except

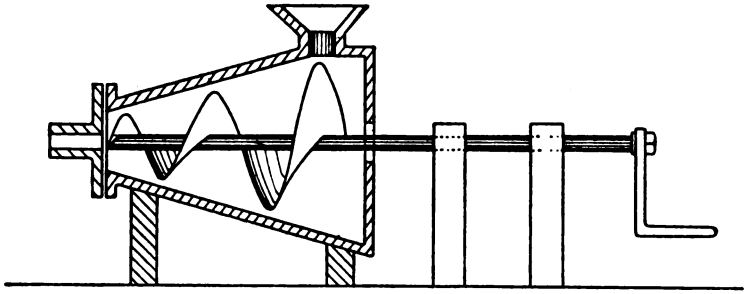

FIG. 19.—Sausage Machine for Gelatinized Explosives.

in the case of very stiff jellies such as Blasting Gelatine, when only one is used.

Gelatinized explosives, like all explosives containing nitroglycerine, have a great tendency to freeze, and in this

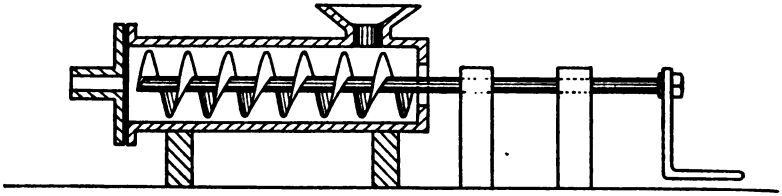

FIG. 20.—Sausage Machine for Gelatinized Explosives.

condition are difficult to detonate. Many accidents have been caused by frozen explosives, as some of the cartridges in the shot hole may escape detonation and remain mixed with the mineral brought down. Also it is difficult to persuade miners to thaw frozen explosives in a warming pan, so that many fatalities have occurred through their being thawed over a fire or by other improper means. Laboratory experiments point to frozen explosives being less

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sensitive to shock than unfrozen ones, but these experiments are quite misleading owing to the small quantities used. Experience under working conditions shows most decidedly that in the frozen state they are more sensitive, and not a few accidents have been caused through miners trying to make a hole in a frozen cartridge in which to insert a detonator.

For this reason many attempts have been made to add substances to the nitroglycerine which will lower its freezing-point sufficiently to allow it to remain liquid at ordinary working temperatures. The freezing-point coefficient of nitroglycerine is 70·5, but Raoult's law only holds good for very dilute solutions, and these are of but little value from a technical point of view.

Non-freezing explosives were originally made by adding nitrobenzole or nitrotoluol to the nitroglycerine, but unfortunately these greatly decrease the power and render detonation more difficult. At present trinitrotoluol is generally used and is frequently applied in the form of the so-called liquid T.N.T., as this mixes more readily with the nitroglycerine than solid T.N.T.

In Great Britain, where very low temperatures are not encountered, the amount used is 5–10 per cent. of the weight of nitroglycerine, but in America and on the Continent 20–25 per cent. is frequently used.

In Germany explosives are made with a mixture of nitroglycerine and dinitroglycerine, tetranitrodiglycerine or dinitrochlorhydrin, and in France dinitroglycol is used. As would be expected from their lower molecular weight, these are more effective than nitroaromatic hydrocarbons. Unfortunately the majority of suitable substances with low molecular weight are too volatile, but nitromethane (B.P. 101° C.) has been proposed, as 10 per cent. reduces the freezing-point of nitroglycerine to −15° C. Its cost, however, is against its use at present.

Dinitroacetyl glycerine and dinitroformin (D.R.P. 209,943) have also been proposed. The latter is very easily made by heating glycerine with half its weight of

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oxalic acid to 100° C. and finally to 150° C., and then nitrating the product. The oil thus obtained contains 33 per cent. of dinitroformin dissolved in nitroglycerine and is almost unfreezable.

The three standard gelatinized explosives in use in Great Britain are Blasting Gelatine, Gelatine Dynamite, and Gelignite, the average composition of which is shown in the following table :—

	N.G.	C.C.	W.M.	KNO_3.
Blasting Gelatine	92	8	—	—
Gelatine Dynamite ..	75	5	5	15
Gelignite	60	4	8	28

Of these Gelignite is far the most used, and may in fact be regarded as the standard high explosive.

Blasting Gelatine, which forms a stiff translucent jelly, is too brisant for most purposes, although used for hard rock. It is the most powerful explosive on the market, but has several serious drawbacks. In the first place it hardens on keeping, and at the same time becomes very insensitive so that it can only be exploded by means of an extra heavy detonator. It also has a great tendency to sweat or exude nitroglycerine, especially if it has been frozen and thawed several times. The sweating is sometimes quite excessive, and the nitroglycerine may actually be seen running out of the cases. Needless to say such Blasting Gelatine is very dangerous. The exudation trouble is very noticeable with explosive that has been exported to the antipodes, and is probably accentuated by the passage through the tropics, hundreds of tons being condemned every year in Australia on account of exudation. It is very difficult to obtain a good Blasting Gelatine that will not sweat and probably most depends on the quality of collodion used. This should contain about 12 per cent. of nitrogen, should have been nitrated at a low temperature and only washed in hot water and not boiled. Finally, it should not have been over-pulped as this, like boiling, adversely affects its gelatinizing powers. Other gelatinized explosives such as Gelatine Dynamite and Gelignite also

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have a tendency to sweat, but these give little trouble as the wood meal holds back the nitroglycerine.

Since 1914, when the outbreak of war made potash supplies scarce, Gelatine Dynamite and Gelignite have been made with sodium nitrate in place of the potassium salt.

Nitroglycerine is slightly gelatinized when used for a large number of explosives, as by thickening it without actually forming a jelly its tendency to be displaced by water is lessened.

In the United States many grades of Gelatin or Gelatin Dynamite are manufactured, in some of which the wood meal is partly replaced by flour and sulphur. They usually contain one per cent. of calcium carbonate, and have a nitroglycerine content of from 20 to 60 per cent.

In France gelatinized explosives are known as Gommes, Dynamites Gélatinés, or simply Gélatinées, and are manufactured in great variety, both potassium and sodium nitrate being used. The following table shows a few typical examples :—

	N.G.	C.C.	KNO_3	$NaNO_3$	W.M.
Gomme extraforte ..	92	8	—	—	—
Gomme B	86	5	4	—	5
Gomme G	83	5	—	10	2
Gomme 3B	74	6	15·5	—	4·5
Gélatinée 1B	64	3	—	24	8
Gélignite	60	3	27	—	10
Dynamite Gélatinée 2C ..	43	2	41	—	4

Dynamite No. 4 and Dynamite No. 5 are both jellies containing sodium nitrate, the former containing 59 and the latter 48 per cent. of nitroglycerine.

The Belgian Forcites are very similar in nature :—

	N.G.	C.C.	KNO_3	$NaNO_3$	W.M.	$MgCO_3$
Forcite extra ..	74	6	14	—	5	1
Forcite supérieure	64	3	—	24	8	1
Forcite No. 1 ..	49	2	—	36	13	—

Some Belgian Forcites also contain ammonium nitrate.

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Chlorate and Perchlorate Mixtures

The formation of chlorates from the metallic chloride and oxygen is an endothermic reaction, and consequently chlorates in themselves are capable of explosion—

$$KCl + 3O = KClO_3 - 11{\cdot}9 \text{ Cal.}$$
$$NaCl + 3O = NaClO_3 - 13{\cdot}1 \text{ Cal.}$$
$$BaCl_2 + 6O = Ba(ClO_3)_2 - 25{\cdot}9 \text{ Cal.}$$

The explosion of a chlorate is brought about by heat, or by heat in conjunction with shock, and although the explosion is usually not violent several serious accidents have occurred. The instability of chlorates renders chlorate mixtures very sensitive, and in most countries the use of chlorates in conjunction with sulphur is prohibited. In Great Britain the only form of chlorate explosive that is authorized is Cheddite, the chlorate in this case being protected and rendered less sensitive by oil. In other countries, however, Sprengel explosives are permitted, in which the chlorate and the combustible matter are only brought in contact with one another immediately before charging the bore hole. Chlorates must never be used in conjunction with ammonium salts, such as ammonium nitrate, as under these conditions double decomposition is liable to take place with the production of the highly unstable ammonium chlorate, a salt liable to spontaneous explosion—

$$KClO_3 + AmNO_3 = KNO_3 + AmClO_3$$

Perchlorates, on the other hand, are much more stable than chlorates, as their formation from the chloride and oxygen is an exothermic reaction—

$$KCl + 4O = KClO_4 + 7{\cdot}9 \text{ Cal.}$$
$$NaCl + 4O = NaClO_4 + 2{\cdot}4 \text{ Cal.}$$
$$BaCl_2 + 8O = Ba(ClO_4)_2 + 4{\cdot}3 \text{ Cal.}$$

Ammonium perchlorate is also stable, but is capable of explosion although only with difficulty. Its detonation is represented by the equation—

$$NH_4ClO_4 = Cl + O_2 + N + 2H_2O \text{ (gaseous)} + 38{\cdot}3 \text{ Cal.}$$

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It is about as sensitive to blows as picric acid, but the explosion does not usually spread throughout the mass.

Chlorates are invariably manufactured by the electrolysis of chlorides, and on further electrolysis pass into perchlorates. For this reason commercial perchlorates are liable to be contaminated with chlorate, and such contamination renders them quite unsuitable for use in the manufacture of explosives. This is particularly so in the case of ammonium perchlorate, as the presence of sodium or potassium chlorate would render the product liable to double decomposition with the production of ammonium chlorate.

Of the chlorates the potassium salt is the most used, as the sodium salt is very soluble and deliquescent and consequently difficult to obtain pure. The latter, however, enters into the composition of some French Cheddites, and barium chlorate is used to some extent as an ingredient in fireworks. Of the perchlorates the almost insoluble potassium salt is most used, although the ammonium salt can be obtained pure at a reasonable price.* This latter salt has scarcely received the attention it deserves in this country, but now that it has been manufactured for war purposes it will probably receive greater attention. It has the advantage of giving only gaseous products, but unfortunately these include chlorine. Chlorine fume can be prevented by adding alkali nitrate in sufficient quantity to provide a base to combine with the chlorine but such mixtures are liable to deliquesce owing to the formation of ammonium nitrate and alkali perchlorate by double decomposition.

$$AmClO_4 + KNO_3 = KClO_4 + AmNO_3$$

For Permitted Explosives, however, the base might be added in the form of an oxalate, this acting at the same time as the "cooler," or the base might be added in the form of a salt of a suitable organic acid, the organic part of the molecule providing the requisite combustible matter.

* In 1918 the cost of manufacture of ammonium perchlorate was £99 per ton as compared with £45 per ton for ammonium nitrate (*C.T.J.*, 1919, p. 162).

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Sprengel Explosives.—These consist of cartridges of potassium chlorate, which immediately before use are dipped into a liquid combustible. They are not authorized in Great Britain, as the process of dipping is a manufacturing operation within the meaning of the Act and can therefore only be carried out on licensed premises and under "danger" conditions. These explosives are used, however, to a considerable extent on the Continent and in America, but have the disadvantage that the penetration of the combustible liquid is very uneven, so that uncertain results are obtained. In France "Explosif O3" or "Promethée" is much used, and consists of cartridges containing 80, 90 or 95 per cent. of potassium chlorate mixed with 20, 10 or 5 per cent. of manganese dioxide, and dipped into a mixture containing 50 per cent. of nitrobenzole and 50 per cent. of turpentine or naphtha, or a mixture of these. The American explosive, Rack-a-Rock, is very similar.

Liquid air explosives, "Oxyliquits," may be mentioned at this point as they are of the Sprengel type although containing no chlorate. They consist of a solid organic absorbent such as cork dust, on which liquid air or oxygen is poured just before use. They were used in the construction of the Simplon tunnel, and are said to have been widely used in Germany for general blasting purposes during the war, but are not likely to come into general use, as liquid air has obvious disadvantages and inconveniences. Also the density of the explosive is low so that the cost of making bore holes is excessive. Liquid air, however, is cheap, especially where extensive mining operations are in progress and large quantities of explosive required. The great advantage of liquid air explosives lies, perhaps, in the absence of danger from misfires or incomplete detonation. With ordinary explosives accidents due to misfires necessitating the withdrawing of the charge or insertion of a new detonator are not uncommon, and incomplete detonation in which cartridges or parts of cartridges remain in the debris are apt to lead to accidents through being struck with a pick or shovel. With liquid air explosives these dangers

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do not exist, as the air rapidly evaporates. They are not authorized in Great Britain, for the same reason that chlorate explosives of the Sprengel class are not authorized.

Cheddites.—These were discovered by Street, and derive their name from Chedde, the town in France where chlorates were principally manufactured. They consist usually of potassium chlorate mixed with a fatty oil, usually castor oil, which has been thickened by dissolving aromatic nitro-compounds in it, although some French Cheddites are made with paraffin. As nitro-compounds a mixture of mononitronaphthalene and dinitrotoluol is generally used, and although the original Cheddite contained 2 per cent. of picric acid this was soon replaced by dinitrotoluol. In their manufacture the nitro-compounds are first dissolved in the oil at a temperature of 70° C. and the finely ground dry chlorate added slowly. The whole is well mixed by hand, first hot and then in a semi-cold condition, sifted and then compressed into cartridges in wooden moulds. In order to obtain a satisfactory explosive the degree of compression must be carefully controlled, especially as Cheddites are usually rather easily compressed. If the density is too low full power is not developed, whereas if too high detonation becomes difficult or impossible. For this reason considerable care is required in making up the cartridges, as some Cheddites are very readily compressed, especially those made with sodium chlorate. The following table shows the velocity of detonation of three different Cheddites at different densities, the experiments being carried out in copper tubes 22 mm. in diameter. The explosives used had the composition—

	Type 60[4].	P.	S.
$KClO_3$	75	90	—
$NaClO_3$	—	—	89
M.N.N.	1	—	—
D.N.T.	15	—	—
Paraffin	—	10	11

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TYPE 60[4]

Temperature.	Density.	Velocity. Metres per sec.
3°–4° C.	·81	2457
	1·39	3045
	1·48	3156
	1·51	3099
	1·62	2820

EXPLOSIF P

Temperature.	Density.	Velocity. Metres per sec.
3° C.	·62	2137
	1·00	3044
	1·05	3185
	1·36	3621
	1·48	3475
	1·54	*Incomplete*
15° C.	·99	2940
	1·24	3457
	1·45	3565
	1·59	*Incomplete*

EXPLOSIF S

Temperature.	Density.	Velocity. Metres per sec.
17° C.	·81	2191
	·92	2475
	1·33	2966
	1·45	2940
	1·54	2688
	1·56	*Incomplete*

The density acquired by the same explosives under different pressures is shown in the following table, the pressure being given in kilograms per cm.[2] :—

Type 60[4].		Explosif P.		Explosif S.	
Pressure.	Density.	Pressure.	Density.	Pressure.	Density.
·7	1·25	·7	·9	·02	·92
1·4	1·31	1·6	·99	·3	1·29
1·6	1·38	5·9	1·21	·6	1·33
2·1	1·40	8·0	1·29	·7	1·39
3·2	1·46	20·0	1·37	1·4	1·47
4·8	1·51	58·0	1·59	3.2	1·55

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The following table shows the composition of the chief French Cheddites :—

	$KClO_3$.	$NaClO_3$.	Castor oil.	M.N.N.	D.N.T.	Paraffin.
Type 60 ..	80	—	6	12	2	—
Type 41 ..	80	—	8	12	—	—
O_2	79	—	5	1	15	—
O_4	90	—	—	—	—	10
O_5	—	79	5	—	16	—

Of these O_5 is the most brisant and O_3 the least.

Cheddites are also authorized and used in Great Britain, but the demand is not great. Thus in 1910, out of a total consumption of 15,000 tons of blasting explosives of all classes, only 60 tons were Cheddite.

In Germany mixtures of chlorates and resin are used to some extent, *e.g.* Silesia No. 4 consists of 80 per cent. of potassium chlorate and 20 per cent. of resin. Some years ago attempts were made to introduce similar explosives into Great Britain under the name of Steelite. These were composed of potassium chlorate and resin which had been previously oxidized by nitric acid. They never came into general use, although Colliery Steelite was on the old "Permitted" list, but failed at the Rotherham test. It had the composition—

$KClO_3$	72·5–75·5
Oxidized resin	23·5–26·5
Castor oil	·5–1
Moisture	0–1

Perchlorates have been suggested as a substitute for nitre in black powder, but have not been used to any extent. A British explosive, Polarite, was introduced some years ago as a substitute for Gelignite and seemed to consist of a non-freezing Gelignite containing T.N.T. in which potassium perchlorate was used in place of nitre. The German explosive, Permonite, was on the old "Permitted" list in Great

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Britain, but failed to pass the Rotherham test. It had the composition—

$KClO_4$	31–34
N.G.	3–4
C.C.	·5–1
$AmNO_3$	39–43
T.N.T.	11–13
Starch	5–9
W.M.	1·5–3·5
Glycerine / Gelatine	1·5–3·5

The French Government experimented with two Cheddites made with ammonium perchlorate and having composition—

	No. 1.	No. 2.
$AmClO_4$	82	50
D.N.T.	13	15
$NaNO_3$	—	30
Castor oil	5	5

but did not authorize them as they presented no advantages over existing Cheddites. The use of castor oil, however, in the presence of perchlorate would seem useless as, unlike the chlorates, they are not sensitive to friction.

The Belgian Yonckites are ammonium perchlorate explosives for use in coal mines, and consist of ammonium perchlorate mixed with sodium and ammonium nitrate, trinitrotoluol and sodium chloride.

The Swedish explosive, Blastine, is composed of ammonium perchlorate, sodium nitrate, D.N.T. and paraffin. Pernitral was the name of an explosive authorized in this country a few years ago, but the manufacture of which does not seem to have been taken up. It had the composition—

$AmClO_4$	40
$NaNO_3$	30
Solid T.N.T.	10
Liquid T.N.T.	10
Al powder	10

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Samples tested by the author seemed to be somewhat more powerful than Blasting Gelatine, but unfortunately a No. 8 detonator was required in order to obtain the full effect.

Ammonium Nitrate Explosives

Explosives consisting of ammonium nitrate in conjunction with combustible matter, a nitroaromatic hydrocarbon for preference, were first proposed by Favier in 1885. They have the advantage of being cheap and safe to manufacture and use, but have the serious disadvantage of being very hygroscopic, so that even when put up in carefully waxed cartridges they take up moisture rapidly and become unfit for use in a few weeks. As a class they have extremely low temperatures of explosion, and for this reason are largely used as coal mine explosives (see Section V.), but are also used to some extent for general blasting purposes. Their use for general purposes is likely to extend in the near future owing to the low price at which they can be manufactured. The best-known British explosives of this class are discussed in the next section, but the following have been used to a considerable extent in Germany—

	Astralit.	Fulmenit.
$AmNO_3$	81	82·5
T.N.T.	11	11
Paraffin Oil	1	1
Meal	2	—
Coal	1	—
N.G.	4	—
Charcoal	—	1·5
Guncotton	—	4

Westfalit für Kohle.

$AmNO_3$	91
KNO_3	4
Resin	5

For ordinary blasting purposes it has been proposed to increase the power of ammonium nitrate explosives by the

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addition of aluminium powder, the heat of combustion of which is very high—

$$2Al + 3O_2 = 2Al_2O_3 + 760 \text{ Cal.}$$

The best known of these explosives is Ammonal, which for ordinary blasting purposes contains about 25 per cent. of aluminium powder, the balance being ammonium nitrate and T.N.T. Ammonals containing much less aluminium, however, are also manufactured, for example—

$AmNO_3$	94	93	88	81
Al	3	3	8	15
C	3	—	4	4
T.N.T.	—	4	—	—

Gesteins-Westfalit contains dinitrotoluol, and has the composition—

$AmNO_3$	84·5
Al	3·5
D.N.T.	12·0

In all explosives of this nature the aluminium is added in the form of a fine powder, but too fine a state of subdivision is to be avoided, as very finely powdered aluminium oxidizes readily in the air. As a rule the commercial "aluminium bronze" is used. This is manufactured by rolling aluminium into very thin sheets, which are then cut into strips and ground.

Very similar explosives in which calcium silicide is used in place of aluminium are also manufactured, Sabulite being an explosive of this class. The use of ferrosilicon has also been proposed (A.P. 1,277,043), although it does not seem to have come into use.

In making up ammonium nitrate explosives into cartridges two methods are used. If loaded by hand the paper is rolled on a wooden rod of suitable size and one end folded in and closed. The paper cylinder thus formed is then inserted closed end downwards in an aluminium tube fixed in a block of wood, and the explosive fed in through an aluminium funnel and well rammed with a wood or aluminium

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rod. This method has little to recommend it except that the plant can be bought for a few shillings. A much more satisfactory method is to use a helix machine very similar to the "sausage" machines used for gelatinized explosives, the machine being made to work against a suitable resistance in order to pack the powder evenly and firmly. A simple machine of this nature for use with hand power is shown in Fig. 21. The explosive is fed in through the hopper and the

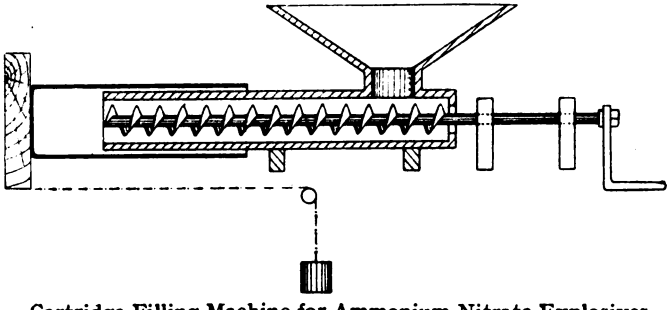

FIG. 21.—Cartridge Filling Machine for Ammonium Nitrate Explosives.

paper cylinder held against the nozzle by means of a cord and weight. As the loading proceeds the paper is forced backwards away from the nozzle, an adjustable stop being provided to indicate when loading is complete. By varying the weight the explosive can be packed more or less tightly at will. In Germany similar machines are used that are power-driven, but these as a rule are vertical.

TONITE

This was formerly much used for blasting purposes, and consisted of compressed guncotton containing from 25 to 40 per cent. of a nitrate, usually barium or potassium nitrate. Its interest is now chiefly historical.

LITERATURE

DYNAMITE

Two interesting accidents which have occurred in the manufacture of Dynamite are described in *S.R.*, 145, 184.

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Gelatinized Explosives

The following accounts of accidents are instructive: *S.R.*, 151, 201.

For notes on the exudation of nitroglycerine from Blasting Gelatine, see *A.E.*, 1914.

Chlorate Explosives

Much information on the properties of Cheddites will be found in *P.S.*, xi., 22; xii., 123; xiii., 29, 144, 282; xiv., 26, 33, 192; xv., 135, 212, 247; xvi., 66.

Two accidents in this country are described in *S.R.*, 135 and 185.

Ammonium Nitrate Explosives

E.P. 2139[05]; 16,277[00]; A.P. 1,277,043.

Blasting

O. Guttmann, "Blasting," London, 1906.

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Section V.—SAFETY COAL MINE EXPLOSIVES

The use of explosives in coal mines is always attended by the risk of firing the mine gases and coal dust. This ignition may be due to some extent to the vibration set up by the shock of the explosion, but the greatest risk is due to local rise in temperature caused by the flame of the explosion or by the adiabatic compression of the gas. Air, for example, when adiabatically compressed to 60 kg. per cm.^{2} suffers a rise in temperature of 670° C., whereas at 100 kg. per cm.^{2} it is 820° C., and at 200 kg. per cm.^{2} it is 1060° C. A mixture of air and fire damp will inflame at 650° C., but at this temperature there is a period of induction of about 10 seconds, although this period grows progressively shorter with increasing temperature. Under working conditions in a mine rapid diffusion takes place so that although locally the temperature may rise far above the point of inflammation, the diffusion of the overheated gases cools them before the period of induction is complete, and hence no explosion results. Of course, there is a limiting temperature above which the period of induction is so short that inflammation takes place before diffusion has had time to lower the temperature below the ignition point, and for safety it is consequently necessary to select explosives and conditions of shot firing in such a way that the local rise in temperature is not excessive. The problem is a difficult one, as mine conditions are very variable, and the chief danger arises from abnormal cases, such as blown-out shots. Consequently the tests applied are of an empirical nature, although designed as far as possible to represent actual working conditions. Usually they imitate what might happen in a mine under the worst possible conditions.

In order to investigate the problem and lay down rules

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for blasting in gassy mines, almost all civilized countries in which coal mining is an important industry have appointed Commissions. The first of these was appointed in France in 1877, the British Commission being appointed two years later, and the Prussian Commission in 1881. The Belgian and Austrian Commissions were both set up at a rather later date. The result of these Commissions has been the setting up of galleries in which explosives can be fired into a gas mixture, or into air or gas laden with coal dust, and the laying down of regulations limiting the composition of the explosives which may be used, the weight that may be used in any one blast, and the conditions under which blasting may be carried out.

Explosive.		Density.	Velocity of Detonation.	Length of Flame.	Duration of Flame.	After-flame Ratio.
G.P.	KNO_3 75 C. 12 S. 13	1·04	200–300	110	77	1:330
B.G.	N.G. 92 C.C. 8	1·63	7700	224	9·72	1:883
Dynamite	N.G. 75 Guhr. 25	1·58	6818	228	8·31	1:620
Gelignite	N.G. 63·5 C.C. 1·5 $NaNO_3$ 27 W.M. 8	1·67	6210	150	1·23	1:101
Donarite	$AmNO_3$ 80 N.G. 3·8 C.C. ·2 T.N.T. 12·0 Flour 4·0	1·31	3930 4137 (confined)	69	·40	1:15
Ammon-Carbonite	$AmNO_3$ 80·3 KNO_3 5·0 N.G. 4·0 C.C. ·2 Coal 6·0 Starch 4·5	1·11	1753 3195 (confined)	51	·32	1:7·4
Thunderite	$AmNO_3$ 92 T.N.T. 4 Flour 4	1·07	2137 3654 (confined)	—	·33	—

Bichel has investigated the duration of flame by firing explosives at night and photographing the flame through a quartz lense on a moving film. He finds that in all cases the flame outlasts the time of detonation, and has named the

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ratio duration of detonation : duration of flame the "after-flame ratio." He finds that safety explosives have a very short flame duration, and consequently a high after-flame ratio, the table in the preceding page showing some of his figures, velocity of detonation being given in metres per second, duration of flame in thousandths of a second and length of flame in millimetres. The experiments were carried out with 100 grams of explosives made up in cartridges of 30 mm. diameter, and fired from a gun similar to those used in the official testing galleries.

In the American testing station at Pittsburg several explosives were analyzed and the flame from 200 grams photographed, and the heat liberated measured. The following very interesting figures were obtained. The heat liberated is expressed in major calories per kilogram, and the duration of flame in milliseconds—

Composition.	Weight of wrapper per 100 grams.	Per cent. CO in products of Explosive.	Heat liberated.	Duration of Flame.
D.N.T. 17·85				
M.N.N. 1·30				
Castor oil 5·32	5	41	1065	·196
$KClO_3$ 75·36				
H_2O ·17				
N.G. 8·13				
M.N.T. 3·57				
Castor oil ·81				
N.C. ·56	6·5	5·9	1169	·279
$AmNO_3$ 82·11				
W.M. 4·30				
H_2O ·52				
KNO_3 65·31				
C. 19·52				
S. 2·63	None	40	622·7	1030
Paraffin 3·35				
Starch 8·73				

This last explosive would appear to be Bobbinite, and in view of its great flame duration it is scarcely surprising that it does not pass the Rotherham Test.

Will has also studied the duration of flame by photographic methods. He took his photographs on a drum

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covered with a sensitive film over which was laid an opaque screen, in which a series of equidistant slits were cut, the whole being rapidly rotated behind a fixed opening. By this means he obtained a species of cinematograph photographs in the form of a series of bands. He found that with explosives deficient in oxygen the flame dies away very rapidly and then revives, the secondary flame being of much greater duration than the primary flame. This secondary flame is due to the carbon monoxide escaping from the bore hole and burning in contact with atmospheric oxygen, and can be prevented by the addition of a few per cent. of an alkali salt, the salts of the alkali metals being far more effective than the salts of the alkali earths or of lead.

The first proposal for rendering explosives used in fiery mines safe by reducing the temperature of explosion was due to MacNab, who in 1873 suggested placing a cartridge of water immediately above the charge in the bore hole. This suggestion was soon followed by others, in which wet moss or a jelly containing 90 per cent. of water was substituted for the inconvenient cylinder of water, but none of these met with any great success, partly owing to the tendency of the miners to omit using them, and partly owing to the contents being blown about in large lumps, and not being sufficiently pulverized by the explosion. The next step was the use of salts containing water of crystallization, such as magnesium sulphate, as these could be incorporated with the explosive itself, and this method is still used to a slight extent, although almost all modern safety explosives rely on the chlorides of sodium, potassium or ammonium as "coolers," the cooling being brought about by the heat absorbed by the volatilization, and to some extent also by the dissociation of these salts. Ammonium chloride has the disadvantage that it is apt to spoil the detonation of the explosive, and it must be used in conjunction with at least one equivalent of an alkali nitrate to provide a base with which the free chlorine liberated during the explosion may combine. It also renders the explosive hygroscopic as double decomposition takes place with the production of ammonium nitrate—

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$$KNO_3 + NH_4Cl = KCl + NH_4NO_3$$

A salt containing water of crystallization when used in conjunction with ammonium nitrate is also objectionable, as the nitrate is apt to rob it of its water and to become liquid by solution.

In Great Britain explosives that may be used in fiery mines are known as "Permitted Explosives," in the United States as "Permissible Explosives," in France as "Explosifs de Sûreté," or "Explosifs Anti-grisouteuses," in Belgium as "Explosifs S. G. P." ("Sûre Grisou Poussiéres"), and in Germany as "Wetter- or Wettersichere Sprengstoffe."

Test Galleries.—The *German* gallery is at Gelsenkirchen-Schalke, and is 35 metres long. It is elliptical in section, and has a sectional area of two square metres, the major axis being vertical and 1·8 metres long, and the minor axis 1·35 metres long. Shots are fired into an explosion chamber, formed by partitioning off a length of 5 metres by means of a paper diaphragm, from a gun with a bore 4 cm. in diameter and 70 cm. deep. The gun is inclined at a slight angle, and the shots are fired without any stemming. There is a statutory obligation for owners of fiery mines to use only "safe explosives," but it rests with the owner to decide what explosive is safe. Several private galleries also exist, notably the one at Neubabelsberg.

The *Austrian* gallery is 293 metres long, and is part of an abandoned mine. The shots are fired suspended in the mine, and not from a gun, thus representing more or less the conditions of an explosive exploding accidentally before being placed in the shot hole.

The *Belgian* gallery at Frameries, near Mons, is similar to the German gallery described above, but is 85 metres long. The shots are fired unstemmed from a gun with a bore 5·5 cm. in diameter and 46 cm. deep, the gun being inclined at an angle. The explosive gas used is air containing 8 per cent. of fire damp, the mixture being warmed to 25° C. The power of the explosive is determined by comparison with Dynamite No. 1 in the Trauzl lead block, and

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in order to pass the test ten shots of an amount equivalent to 175 grams of Dynamite must fail to fire the gas mixture, and also fail to fire coal dust. The largest charge that satisfies these conditions is the "charge limite" or the maximum amount of that explosive that may be used in any one blast.

The *French* galleries are at Liévin, one being 15 metres long and 2 metres in area, and the other 300 metres long and 2·8 metres in area, both being of circular section and supplied with natural gas. The French regulations, however, are not based on experiment, but rather on theoretical considerations. For use in coal mines an explosive must on detonation yield no combustible products such as carbon monoxide, and for use in rock (explosif roche) its calculated temperature must not exceed 1900° C., or for use in coal (explosif couche) 1500° C. These calculated temperatures are based on the heat of combustion of the constituents and on the specific heat of the products of combustion, and further details will be found in Section VIII.

The *United States* gallery is situated at Pittsburg. It is 100 ft. long by 6 ft. 4 in. in diameter, and is constructed of boiler plate, and closed at one end by concrete. Safety doors are placed along the top to prevent undue pressure being developed. Shelves are placed along the sides of the gallery to hold coal dust, this dust being obtained from bituminous coal ground until it passes a 100-mesh sieve.

The shots are fired unstemmed from a gun 24 in. in diameter and 36 in. long, with a bore hole 2¼ in. in diameter and 21½ in. deep. Natural gas (roughly equal parts of methane and ethane) is used, and the gas-air mixture is warmed to 77° F. Power is determined in the ballistic pendulum (see Section VIII.) and is compared with American 40 per cent. Straight Dynamite of the composition—

N.G.	40
$NaNO_3$	44
W.M.	15
$CaCO_3$	1

In order to pass the test and be placed on the list of

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"Permissible Explosives," an explosive must fulfil the following conditions :—

1. Ten shots of a weight equivalent to ½ lb. of the standard 40 per cent. Dynamite fired into 8 per cent. of gas must cause no explosion.

2. Ten shots of the same weight fired into 4 per cent. of gas with the addition of 20 lbs. of coal dust must cause no explosion.

3. Ten shots of the same weight fired into 40 lbs. of coal dust must cause no explosion.

The limit-charge is determined by firing amounts increasing by 25 grams into 4 per cent. of gas and 20 lbs. of coal dust until the maximum amount is found of which 10 shots can be fired without causing an explosion. The composition of explosive is not published.

The first *British* gallery was built at Woolwich, and was only 27 ft. 6 in. long by 2 ft. 6 in. diameter. The explosives were fired from a gun with a bore hole 30 in. deep and 1⅞ in. diameter, and were stemmed with a definite weight of dry clay. The explosive mixture used was air containing 15 per cent. of coal gas. This gallery has been abandoned since 1912, in which year the Rotherham gallery came into use. This is 50 ft. long by 5 ft. in diameter, and is constructed of ½ in. mild steel plate. The explosion chamber is 18 ft. long, and is separated from the rest of the gallery by a paper diaphragm. The outer end is closed by a ¾-in. steel plate, a hole being provided for inserting the gun and arrangements made for a gas-tight joint. The explosion chamber has three windows made of ¾-in. plate glass, each being 6 in. square. There are nine pressure release valves on the top.

The gas is measured in a gas holder, and then made to circulate with the air in the explosion chamber by means of fans, complete mixing being brought about in 3 minutes. The mixture used contains 13·4 per cent. of gas.

The gun is of steel (wire construction), with a bore hole 55 mm. in diameter and 120 cm. deep.

The following are the conditions regulating the test as abstracted from A.R. 1912 :—

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The explosive must be on the List of Authorized Explosives. The Secretary of State may at any time cause any explosive on the Permitted List to be formally retested. Notice of any such formal retest will be sent to the manufacturer of the explosive.

For the test the following weights and sizes of cartridges must be supplied :—

33 lbs. $1\frac{3}{8}''$ × 8 oz.
10 lbs. $1\frac{3}{8}''$ × 4 oz.
5 lbs. $1\frac{3}{8}''$ × 2 oz.
2 lbs. $1\frac{3}{4}''$ × 4 oz.

The Secretary of State reserves to himself the right of storing all explosives submitted for the Official Test for at least 30 days prior to the test.

The fees for testing an explosive are :—

Testing a new explosive	£50
Testing an altered explosive	£30
Each experimental shot	£1

These experimental shots have no official significance, but are for the benefit of manufacturers in order to enable them to form some idea as to whether an explosive is likely to pass or not before paying the somewhat heavy fee for the Official Test.

The Official Test is carried out as follows (official description) :—

" Shots will be fired into a mixture of gas and air until the largest charge which can be fired without igniting it is found. Further shots will then be fired, beginning with this charge and, in the event of an ignition, reducing the charge—until five shots of the same weight have been fired without igniting the mixture. Shots will then be fired with the charge so determined into coal dust, and the same procedure adopted until 5 shots have been fired without igniting the coal dust. The lower of the charges thus determined will be known as the ' maximum charge.' In making alterations

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to the weight of the charges, the increment or decrement of charge will not be less than two ounces."

"In loading the gun the charge will be pushed to the bottom of the bore (a clay plug having been previously inserted to protect the crown of the bore), and will have no tamping. The coal dust will be ground to such a degree of fineness that not less than 90 per cent. will pass through a sieve of 150 meshes to the linear inch."

"In addition to the foregoing shots, other shots will be fired at the ballistic pendulum, and the swings registered on the sliding scale provided for the purpose will be recorded. The mean swing thus obtained will be published in comparison with that given by a charge of Gelignite containing 60 per cent. of nitroglycerine."

"Every shot will be fired electrically, and in the case of high explosives a detonator of the size recommended by the manufacturer or the person submitting the explosive will, subject to the approval of the Home Office, be used."

"Each shot will be fired in the case, wrapper or covering in which the explosive is proposed to be employed in actual use."

"An explosive will be considered to have passed the test if—

"(1) The 'maximum charge' as above determined, is not less than eight ounces.

"(2) If in the shots at the pendulum no appreciable amount of the charge has been left unexploded.

"(3) If, in the opinion of the officer in charge of the testing, the explosive has exploded in a satisfactory manner when fired untamped at the gallery."

"The heaviest charge which may be fired from the gun is 2½ lbs."

The following are extracts from the chief conditions governing the packing and use of Permitted Explosives (Coal Mines Act, Order in Council, Sept. 1st, 1913):—

"No drill shall be used for boring a shot hole unless it allows at least a clearance of ⅛ in. over the diameter of the cartridge which is intended to be used in the shot hole."

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"Every charge shall . . . have sufficient stemming, and each such charge shall consist of a cartridge or cartridges of not more than one description of explosive."

"In all coal mines in which inflammable gas has been found within the previous three months in such quantity as to be indicative of danger, no explosive, other than a Permitted Explosive . . . shall be used."

"In all coal mines which are not naturally wet throughout no explosive, other than a Permitted Explosive . . . shall be used . . . in any road or any dry and dusty part of the mine."

The following regulations must be followed as regards packing and marking, and are in addition to those made under the Explosives Act, 1875 :—

Every outer package must bear the words "As defined in the List of Permitted Explosives."

Each inner package must bear the words "Permitted Explosive, to be used only with not less than No. — detonator," the number of the detonator being that which is given in the Permitted List. It must also be marked with the name of the explosive, the name of the manufacturer, the place and date of manufacture, and the nature and proportion of the ingredients as set forth in the Permitted List.

Each cartridge must be stamped with a P set in a crown, and must bear the words "Not more than — ounces in any one shot hole," the number of ounces being the maximum charge as determined by the Official Test.

In the Permitted List each explosive is defined as consisting of certain ingredients, a maximum and minimum figure being given for each to allow for manufacturing error. This allowance, which is fixed by the manufacturer with the approval of the Home Office, is frequently absurdly high. For example, Super-excellite No. 2 is defined as containing not more than 6 or less than 4 parts by weight of nitroglycerine. In making up a 500-lb. batch with the mean value, 5 parts, 25 lbs. of nitroglycerine would be required, but an error of 5 lbs. in either direction might be made without

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departing from the definition. Needless to say an error of 5 in 25 is absurd. A liberal allowance with a hygroscopic substance like ammonium nitrate is more sensible, but even here the allowance is decidedly high. These high allowances for error mean that a manufacturer could make an explosive with a number of ingredients and submit a sample to the Official Test in which the inert matters were kept at their maximum and the active ingredients at a minimum. After having passed the test he could then reverse the process and sell an explosive in which the inert material was at a minimum, and the active ingredients at a maximum, although this actual mixture might not have passed the test if submitted.

The Permitted List also states the name of the manufacturer, and the situation of the factory in which the explosive must be made, the smallest detonator which may be used to fire the explosive and the greatest weight that may be used in any one shot hole. It states the nature of the wrapper, *e.g.* parchment paper, tin-lead alloy cases, etc., and in the case of waterproofed cartridges states the nature of the waterproofing material, *e.g.* paraffin wax, carnauba wax, wax and resin, etc. It does not, however, set any limit to the relative weight of the wrapper, although this is considerable, especially in small cartridges, and when of a combustible nature forms a part of the explosive. However, the weight of the wrapper would be difficult to fix, as it varies so much with the different sizes and, possibly, is not of such importance as it would appear to be. For example, Ammonite No. 1 and Ammonite No. 5 have the same composition, viz.—

$AmNO_3$	73–77
T.N.N.	4–6
NaCl	19·5–21·5
H_2O	0–1

but No. 5 is made up in waxed paper cartridges, whereas No. 1 is made up in cases "of lead and tin alloy thoroughly waterproofed with pure paraffin wax." The former gives

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a swing of 2·41 in. with the ballistic pendulum and has a maximum charge of 26 oz., whereas the latter gives a swing of 2·42 in., and has a maximum charge of 24 oz. These figures are very close, but it must be borne in mind that both are stated to be waxed, and that Ammonite contains a very large excess of oxygen over and above that required for the complete combustion of the trinitronaphthaline present. The case might be quite different with those explosives which contain only just sufficient oxygen for complete combustion, as the waxed wrapper would then cause the production of large quantities of carbon monoxide.

In order to examine the influence of the wrapper two series of experiments were carried out in the Belgian galleries. In the first series Grisounite roche, an explosive containing a considerable excess of oxygen and having the composition—

$AmNO_3$	91·5
D.N.T.	8·5

was used, and the combustible gas in the products of combustion estimated. The results obtained were striking—

Wrapper.	Per cent. combustible gas.
None	2·4
Asbestos paper	4·0
Paraffined paper	20·0
Aluminium or tinfoil	2·0
Paraffined paper plus 20 grams coal dust ..	37·5
Tin foil plus 20 grams coal dust	55·6

The last figure is rather extraordinary and seems difficult of explanation.

In the second series of experiments various Belgian coal mine explosives were fired in the gallery with different wrappers, and the charge limite determined. In all cases the use of paraffined paper in place of unparaffined paper lowered the charge limite to a very marked extent, in some cases by as much as 85 per cent., both when fired into gas and when fired into coal dust. Metal foil also reduced the charge limite but to a lesser extent, this reduction being

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probably due partly to the great heat of combustion of the metal. The substitution of asbestos paper for ordinary paper, on the other hand, was found to raise the charge limite by as much as 50 per cent. From this it is obvious that the effect of the wrapper is very great, but no data are available as to the influence of wrappers on different sizes of cartridges, nor do any experiments seem to have been carried out in this country.

In gallery tests the diameter and length of the bore hole of the gun used affects the test to a marked extent, and experiments have been carried out in Germany on these lines. Two guns were used, one having a bore 55 mm. in diameter and 57·5 cm. long, and one having a bore 40 mm. in diameter and 70 cm. long. It was found that when fired from the 55 mm. gun cartridges of 35 mm. diameter gave decidedly higher maximum charges when fired into gas than cartridges of 55 mm. diameter, whereas with coal dust there was but little difference. On the other hand, 35 mm. cartridges fired from a 40 mm. gun gave slightly higher charges both in gas and in coal dust than did 40 mm. cartridges.

The length of the bore hole has also a considerable influence, the longer the hole the bigger the maximum charge obtained, but this is probably due to the increased cooling surface of the metal. The influence of the magnitude of the sectional area of the gallery has also been studied, and a decreasing area is accompanied by a decreasing charge limite, but results are very irregular. In some cases the reduction is greatest when the explosive is fired into gas, and in others the reduction is greater when coal dust is used.

No gallery test can be said to represent the conditions actually attained in mining practice, but they all err on the safe side by providing for conditions more dangerous than are likely to be met with. Tamping has a great influence, and the absence of tamping makes the gallery tests much more severe, whereas in blasting, of course, shots are never fired without stemming.

As regards the British test, this differs from the Belgian and American tests by not specifying any temperature for

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the gas in the explosion chamber, although workings are frequently decidedly warm. Another point which seems to be completely overlooked is barometric pressure. Some workings are several thousand feet deep, and as the barometric height increases by about $\frac{1}{10}$ in. for every hundred feet descended, the increased pressure in these workings is considerable. Surface variations of barometer in this country do not, as a rule, exceed 1 in., corresponding to a depth of 1000 ft., but pending properly constituted experiments it would be an advantage if the barometer reading and temperature of the gas at the time of the test was stated in the Permitted List. Owing to the slight variations of barometric pressure experienced the information might not be of great value, but the trouble would be so slight that it would be worth doing. As will be seen from the above remarks, a vast amount of experimental work should be carried out on the use of explosives in the presence of inflammable gas mixtures and coal dust. So far no work at all seems to have been done in this country, except the official testing of explosives for manufacturers. Owing to the expense it is hardly work that a private individual, or even a company, would care to take up, and from the national importance of the subject it is decidedly one that should be taken in hand by the Government. The Rotherham station has now been in use for six years, but so far there seems to have been no attempt made to carry out any research. The subjects that particularly require investigating are: (1) the influence of the wrapper; (2) the influence of barometric pressure; (3) the relative efficiency of the various "coolers"; (4) temperature of explosion; (5) duration of flame; (6) velocity of detonation; and (7) the diameter of the cartridges.

British Permitted Explosives.—The number of explosives on the British List of Permitted Explosives is now (1919) 77. Of these all the non-hygroscopic explosives are put up in cartridges of parchment paper, whereas with one or two exceptions the hygroscopic explosives are put up in paper cases made thoroughly waterproof with a wax such as paraffin, carnauba or ceresin wax, or with a mixture of wax

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and resin. Of the exceptions Stanford Powder, Ammonite No. 1 and Ammonite No. 4 are put up in cases made of lead and tin alloy, thoroughly waterproofed with wax; and Abelite No. 4 is put up in a waxed paper case "with or without an additional covering of tin foil." A German explosive, Tutol No. 2, which was on the list at the outbreak of the war, was put up in a non-waterproofed wrapper of parchment paper and an outer wrapper of waterproofed paper, this outer wrapper being removed before charging the shot hole. This was an obvious attempt to get over the influence of the wrapper as far as possible, and although the explosive is no longer on the Permitted List it is worth giving its composition—

N.G.	24–26
$NaNO_3$	28–30
NaCl	8·5–10·5
W.M.	31–34
$NaHCO_3$	0–·5
H_2O	2·5–5

It gave a swing at the ballistic pendulum of 2·11 in., as compared with 3·27 in. given by an equal weight of Gelignite containing 60 per cent. of nitroglycerine, and the maximum charge was 22 oz.

It is difficult to classify the explosives on the list, but, roughly, they fall into three groups, viz. (1) those containing no nitroglycerine, but consisting of a nitroaromatic hydrocarbon in conjunction with ammonium nitrate with or without sodium or potassium nitrate; (2) those containing nitroglycerine and combustible matter in conjunction with a nitrate; (3) those containing potassium perchlorate.

It is noticeable that the only explosives containing barium nitrate are those rich in nitroglycerine (20–30 per cent.) and containing no nitroaromatic compound, and that all the perchlorate explosives are also rich in nitroglycerine (20–40 per cent.) except Sunderite, which contains only 8–10 parts.

Of the "coolers," the chlorides of sodium, potassium and ammonium are all used, of which the potassium salt seems to be the most effective. Ammonium chloride tends to spoil

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detonation, and in any case can only be used in conjunction with sodium or potassium nitrate, as a fixed base must be provided with which the chlorine liberated can combine. Ammonium oxalate is also used as a cooler, but its use up to the present is confined to explosives containing nitroglycerine. It is present in large quantity, 25–40 per cent., in all the perchlorate mixtures except Samsonite No. 2.

The most powerful explosive on the list is Victor Powder, containing about 9 per cent. of nitroglycerine and giving a swing of 2·96 in., the next most powerful being Nationalite No. 1, containing as explosive ingredients only T.N.T. and ammonium nitrate, and giving a swing of 2·91 in.

No attempt will be made to enumerate all the explosives on the list, but the following tables give some of the chief ones arranged for purposes of comparison. In all cases, the "swing" is that given at the ballistic pendulum by 4 oz., and is to be compared with the swing of 3·27 in. given by the same weight of Gelignite containing 60 per cent. of nitroglycerine.

The mean value of the maximum and minimum parts by weight as set forth in the List of Permitted Explosives is given in all cases.

EXPLOSIVES CONTAINING NO NG.

Explosive.	T.N.T.	D.N.B.	$AmNO_3$.	KNO_3.	$NaNO_3$.	KCl.	NaCl.	NH_4Cl.	H_2O.	Swing.	Max. charge (ounces).
Bellite No. 2 ..	—	12	61	—	—	—	27	—	·35	2·42	32*
Bellite No. 4 ..	—	13	68·5	—	—	—	18	—	·75	2·72	12
Abelite No. 1 ..	6·7	7	68	—	—	—	17·5	—	·5	2·85	14
Nationalite No. 1	15	—	66	—	—	—	19·5	—	·5	2·92	12
Bellite No. 1 † ..	15	—	63·5	—	—	—	16·5	—	—	2·74	20
Nationalite No. 2	15	—	63·5	—	—	21	—	—	·5	2·63	20
Roburite No. 4 ..	16·5	—	60·5	—	—	—	22·5	—	·75	2·86	18
Kentite	15	—	34	33·5	—	—	—	17	1	2·64	18
Anchorite	12	—	34	—	33	—	—	20	·75	2·73	14
Expedite	12	—	34·5	33	—	—	—	20	·75	2·62	32
Denaby Powder ..	13	—	34	33·5	—	—	—	19·5	·75	2·74	18
Super-Curtisite ..	10	—	38·5	29·5	—	—	—	22	1	2·71	16

* This was the largest charge that could be loaded into the gun.
† Contains also 3·5–5·5 parts of starch.

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In the preceding table the following comparisons are interesting. Bellite No. 4 and Abelite No. 1 are almost identical in composition, except that in the latter half the D.N.B. has been replaced by an equal weight of T.N.T. This has increased the power and at the same time raised the maximum charge by 2 oz. In Nationalite No. 1 the substitution has been taken further, with the result that still greater power is obtained, although the maximum charge has fallen back to 12 oz. Bellite No. 1 differs only from Nationalite No. 1 in containing less salt and rather less ammonium nitrate which, surprisingly enough, has caused a falling off in power and an increase in the maximum charge. This may be due to influence of wrapper, or more likely to a different method of incorporating the ingredients. Anchorite and Expedite are very similar in composition, although the maximum charges show a big difference.

The following permitted explosives contain no nitroglycerine, and all contain a very large excess of oxygen over and above that required for the complete combination of the oxidizable ingredients :—

EXPLOSIVES CONTAINING NO N.G. BUT A LARGE EXCESS OF OXYGEN.

Explosive.	T.N.T.	D.N.N.	T.N.N.	$AmNO_3$.	$NaNO_3$.	KNO_3.	NaCl.	KCl.	AmCl.	H_2O.	Swing.	Max. charge (ounces).
Dreadnaught Powder ..	4	—	—	75	15·5	—	—	—	5	·5	2·05	32
Westfalite No. 3	5	—	—	59·5	—	14	—	—	21	·5	2·55	12
Ammonite ..	—	5·5	—	73	—	—	21	—	—	·5	2·44	18
Ammonite No. 4	—	4	—	66	10	—	—	20	—	·5	1·76	30
Ammonite No. 5	—	—	5	75	—	—	20·5	—	—	·5	2·41	26
Ammonite No. 1	—	—	5	75	—	—	20·5	—	—	·5	2·42	24

In the above, Dreadnaught Powder is interesting as having a large maximum charge, although it contains very little cooler. Also the difference in the maximum charges of Ammonite and Ammonite No. 5 is bigger than one would expect from the slight difference in composition. The only difference between the composition of Ammonite No. 1 and Ammonite No. 5 is that the latter is put up in waxed paper and the former in metal foil cases.

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EXPLOSIVES POOR IN N.G.

Explosive.	N.G.	W.M.	$AmNO_3$.	KNO_3.	$NaNO_3$.	NaCl.	KCl.	AmCl.	AmOx.	H_2O.	Swing.	Max. charge (ounces).
Super-Excellite	4·2	3	75·2	7·2	—	—	—	—	10	·75	2·74	10
Super-Excellite No. 2	5	—	50	20	—	—	—	5	15	·75	2·72	14
A 1 Monobel ..	10	—	60	—	—	—	20	—	—	1	2·78	28
A 2 Monobel* ..	10	9	59	—	—	—	20	—	—	1	2·44	22
Thames Powder No. 2	10	9	58·5	—	—	21	—	—	—	1	2·59	22
Viking Powder* No. 1	10	9	59	—	—	19·5	—	—	—	1	2·44	26
Viking Powder* No. 2	8·5	8	67	—	—	15	—	—	—	1	2·59	18
Rex Powder ..	12	7·7	59·5	—	—	19·5	—	—	—	1·2	2·61	20
Stomonal No. 1	10	—	56	—	6	19·5	—	—	—	1	2·68	20
Stomonal No. 2	10	6	61	—	—	17	—	—	6	1	2·57	30
Monobel No. 1 ..	8·5	8	68	—	—	15	—	—	—	1	2·81	10
Victor Powder ..	8·5	8	68	—	—	—	15	—	—	1	2·96	18
Victor Powder No. 2† ..	8·5	8	66·5	—	—	—	15	—	—	1	2·63	16
Du Pont's Permissible No. 1	9·5	7·5	67·5	—	—	15	—	—	—	·75	2·82	18

* Contains in addition ·5–1·5 $MgCO_3$. † Contains in addition 0–2 $MgCO_3$.

Of the powders in the above list A1 Monobel and Stomonal No. 1 both contain a large excess of oxygen, this being remedied in the case of A2 Monobel by the addition of wood meal.

A2 Monobel is almost identical with Viking Powder No. 1, except that in the latter sodium chloride has been substituted for potassium chloride, the result being an increased maximum charge with the same power contrary to what would be expected (cf. Monobel No. 1 and Victor Powder). On the other hand, Thames Powder No. 2 is almost identical with Viking Powder No. 1, but has a smaller maximum charge, although it is more powerful. As these explosives are made by different firms the variation may be due to different mixing or to a different amount of wax on the wrapper.

Victor Powder and Victor Powder No. 2 are very similar, except for an optional 0–2 per cent. of magnesium carbonate in the latter, and the difference in power is quite remarkable. The official definition gives in each case nitroglycerine 7·5–9·5, potassium chloride 14–16, and wood meal

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7–9. For Victor Powder the ammonium nitrate is given as 66·5–69·5, and for Victor Powder No. 2 as 65–68 with magnesium carbonate 0–2. Consequently without departing from the definition the two explosives could be made up to have the same composition, and this serves as a good example of the excessive allowance for manufacturing error permitted in the definition (see p. 126). Du Pont's Permissible No. 1 is the only American made explosive on the British Permitted List at present. It is very similar to Viking Powder No. 2.

Dynobel No. 3 and No. 4 have the following composition, and represent an intermediate stage between the foregoing explosives poor in nitroglycerine and the explosives rich in nitroglycerine :—

	Dynobel No. 3.	Dynobel No. 4.
N.G.	14–16	14–16
C.C.	·25–·75	·25–·75
D.N.B. / D.N.T. / T.N.T.	2·5–5	2–4
$AmNO_3$	51–54	44–47
W.M.	4–6	4–6
NaCl	24–26	28–31
$MgCO_3$	0–1	0–1
H_2O	0–2	0–2
Swing	2·50	2·35
Max. charge	18 oz.	30 oz.

Dynobel No. 2 is similar, but contains more nitroglycerine, 18·5–20·5 parts.

EXPLOSIVES RICH IN N.G.

Explosive.	N.G.	C.C.	W.M.	Starch.	$Ba(NO_3)_2$.	$NaNO_3$.	KNO_3.	NaCl.	AmOx.	H_2O.	Swing.	Max. charge (ounces).
Britonite No. 2 ..	24	—	35	—	—	—	30·5	—	8	3·2	2·26	24
Pitite No. 2 ..	24	—	34·5	—	—	—	29·5	—	8	3·7	2·15	32
Super-Kolax ..	25·5	—	27	7	·	—	25·5	—	7	3	2·10	30
Super-Kolax No. 2	28·5	1	28	8·5	5	—	16·5	—	9·5	3·5	2·21	32*
Cambrite.. ..	23	—	33·5	—	3·7	—	27·5	—	8	4·7	1·98	30
Kynarkite ..	25	—	35	—	3	—	28	—	5	3·7	2·21	20
Britonite No. 3 ..	24·5	—	32	—	—	—	28	12	—	3	2·59	22

* This was the greatest weight that could be loaded into the gun.

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It should be noticed that Britonite No. 2 and Pitite No. 2 are very similar, and the two explosives could be made up to have the same composition without departing from their official definitions, although the maximum charge differs by 8 oz.

It will further be observed that in all these explosives a large quantity of wood meal is used to absorb the nitroglycerine, and that, except in the case of Britonite No. 3, deliquescent salts are not used. This is on account of the danger of water being taken up and displacing the nitroglycerine.

A comparison of Duxite and Arkite No. 2 is very instructive in showing the great difference in maximum charge that can be brought about by a slight difference in composition. Duxite is a German-made explosive that was placed on the British Permitted List in 1914, although it has since been removed. Arkite is made by Kynoch-Arklow, Ltd., and was also placed on the list in 1914, and is still there. The official definitions of these explosives are—

	Duxite.	Arkite No. 2.
N.G.	31–33	31–33
C.C.	·75–1·5	·5–1·5
$NaNO_3$	27–29	—
KNO_3	—	26–28
W.M.	8–10	8–10
AmOx.	28–31	29–31
H_2O	0–2·5	0–2
Swing	2·45	2·41
Max. Charge	12 oz.	40 oz. *

As will be seen, the two explosives are almost identical, except that Duxite contains sodium nitrate in place of potassium nitrate. The enormous difference in the maximum charges is probably due to the wrapper, Arkite No. 2 being put up in parchment paper and Duxite in paper waterproofed with paraffin wax.

The only Permitted Explosive which is a true jelly is Super-Rippite. It is officially defined as follows :—

* This is the greatest charge which may be loaded into the gun.

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N.G.	51–53
C.C.	2–4
KNO_3	13·5–15·5
Borax (dried at 100° C.)	15·5–17·5
KCl	7–9
H_2O (total)	5–8
Swing	2·53
Max. charge	18 oz.

It is put up in non-waterproofed wrappers of parchment paper. Samsonite No. 2 and Samsonite No. 3 are somewhat similar, but the former contains 10–12 parts of potassium perchlorate.

The following explosives containing potassium perchlorate are on the Permitted List:—

EXPLOSIVES CONTAINING PERCHLORATE.

Explosive.	N.G.	C.C.	T.N.T. plus D.N.T.	Starch.	W.M.	$KClO_4$.	AmOx.	H_2O.	Swing.	Max. charge (ounces).
Swale Powder ..	19	1	4	—	9	37·5	28	1	2·50	20
Ajax Powder ..	22·5	·75	3	—	10·5	37·5	25	·75	2·69	12
Neonal	21	1	·2	—	15	37	25	1	2·56	16
Dynobel	32·5	·75	—	—	9·5	27	29·5	·75	2·61	22
Herculite ..	33	1	—	—	9	27	29	1	2·72	16
Neonal No. 1 ..	40	2	—	—	4·5	14	39·5	·5	2·51	30

Among these it is noticeable that Dynobel and Herculite are almost identical in composition and could be made to have the same composition without departing from the official definitions. The difference of the maximum charges is 6 oz., which may be accounted for by a different method of mixing, as the explosives are made by different firms, Dynobel being made by Nobel's Explosives Co., Ltd., and Herculite by the British Explosives Syndicate.

Bobbinite is a non-detonating explosive of the gunpowder type which has failed to pass the Rotherham test, but the use of which is permitted under certain conditions until December 31st, 1919. There are two definitions, of which the second is the most popular—

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	1st Definition.	2nd Definition.
KNO_3	62–65	63–66
C	17–19·5	18·5–20·5
S	1·5–2·5	1·5–2·5
Ammonium Sulphate } Copper Sulphate } ..	13–17	—
Paraffin Wax	—	7–9
Rice or Maize Starch ..	—	2·5–3·5
Moisture	0–2·5	0–3
Density (not exceeding) ..	1·42	1·48

Each pellet must be coated with paraffin wax of a melting point not less than 120° Fahr., and be contained in a wrapper of brown paper. There is no limit to the charge which may be used.

The conditions for its use are as follows:—It may only be used "for bringing down coal (whether by shots placed in the coal or by shots placed in the stratum immediately above or below the coal) and only in . . . mines which are not liable to blowers or sudden outbursts of firedamp, and in which firedamp does not exist in the coal at a pressure which makes the use of such explosives dangerous, and in which the dust . . . is either naturally so largely composed of incombustible matter as not to be dangerous or has been rendered so by artificial means."

"The explosives shall only be used with an electric fuse containing 5 grains of gunpowder or with other means equally efficient in igniting the explosive, but not with a detonator or electric detonator."

German Explosives.—A considerable number of "Wettersichere Sprengstoffe" are available for use in German coal mines, of which the following give a fair idea of the types used:—

Neu Westfalit.		Wetter-Fulmenit.	
D.N.T. ..	10·9	T.N.T. ..	5·5
$AmNO_3$ ..	70·3	G.C. ..	4·0
Flour ..	2·0	$AmNO_3$ ..	75·5
NaCl ..	16·8	C	1·5
Max. charge 540 grams.		Paraffin oil	2·5
		NaCl ..	10·0

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Dahmenit.	
Naphthalene	6·5
$AmNO_3$	91·3
$K_2Cr_2O_7$	2·2

	Dorfit I.	Dorfit II.
T.N.T.	6	15
$AmNO_3$	61	65
KNO_3	5	5
Flour	4	4
NaCl	20	15
Max. Charge	532 grams.	300 grams.

Tremonite S. II.		Gelatin-Carbonit.	
Dinitroglycerol	33	N.G.	25·5
C.C.	1	C.C.	·7
Meal	12	$AmNO_3$	41·5
T.N.T.	2·5	Glycerine and Gelatine	7·0
$AmNO_3$	26·5	NaCl	25·3
NaCl	25		

Gelatin-Wetterastralit I.			
Dinitrochlorhydrin	16	W.M.	·5
N.G.	4	$AmNO_3$	40
C.C.	·5	$NaNO_3$	75
M.N.T.	1	Am. Ox.	2·5
D.N.T.	2	NaCl	14·0
Potato meal	8	Oil	2

This latter explosive probably constitutes a record in complexity of composition.

Austrian Explosives.—The Austrian coal fields are only of small extent, and the manufacture of explosives is a Government monopoly. As a result only two explosives are made for use in coal mines. They are Dynammon and Wetter-Dynammon, and have the composition—

	Dynammon.	Wetter-Dynammon.
$AmNO_3$	87	94
KNO_3	—	2
C	13	4

Belgian Explosives. — A considerable number of

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explosives are on the Belgian List of Explosifs S.G.P., of which the following are typical examples :—

	Dynamite Antigrisouteuse II.	Grisoutite.
N.G.	44	44
W.M.	12	12
$Na_2SO_4.10H_2O$	44	—
$MgSO_4.7H_2O$	—	44
Charge limite ..	650 grams	300 grams.

	Kohlen Carbonite.	Minite.
N.G.	25	25
KNO_3	34	35
$Ba(NO_3)_2$	1	—
Flour	38·5	39·5
Tan Meal	1	—
Na_2CO_3	·5	·5
Charge limite	900 grams	750 grams.

	Permonite S.G.P.	Yonckite 10 bis.
N.G.	6	—
T.N.T.	7	10
$KClO_4$	24·5	—
$AmClO_4$	—	25
$AmNO_3$	29·5	30
$NaNO_3$	—	15
Flour	4	—
Glycerine gelatine ..	3	—
W.M.	3	—
NaCl	24·5	20
Charge limite	900 grams	

A large number of ammonium nitrate explosives are also on the list of explosifs S.G.P., of which the following table gives a few typical examples :—

Explosive.	T.N.T.	D.N.N.	$AmNO_3$.	KNO_3.	$NaNO_3$.	AmOx.	AmCl.	Charge Limite (grams).
Densite 4	19	—	18	45·5	—	—	17·5	850
Densite 3	4	—	74	—	22	—	—	700
Favier 2 bis. ..	—	2·4	77·6	—	—	—	20	500
Fractorite B. ..	—	2·8	75	—	—	2·2	20	450

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A calcium silicide explosive, Sabulite Antigrisouteuse, is also on the list, and has the composition—

T.N.T.	6
$AmNO_3$	54
KNO_3	22
Ca_2Si	5
AmCl	13

French Explosives.—The French explosifs de sûreté, or explosifs antigrisouteuses, are defined by calculation of temperature of explosion, and are divided into two classes, viz. "Grisounites roches," with a temperature of explosion of between 1500° C. and 1900° C., which can only be used in rock, and Grisounites couches with a temperature of explosion below 1500° C. which can be used in coal. The following are a few examples :—

Explosif.	M.N.N.	D.N.N.	T.N.N.	$AmNO_3$.	$NaNO_3$.	KNO_3.
N 1a, bis. couche ..	—	—	5	95	—	—
N 4 couche ..	—	—	5	90	—	5
N 1b, bis. roche ..	—	8·5	—	91·5	—	—
N 1c, bis. roche ..	—	12·6	—	87·4	—	—
N 2 roche	20	—	—	—	80	—
N 3 roche	—	—	27	15	58	—

LITERATURE

Commissions

A summary of the reports of the various commissions first appointed to inquire into the use of Explosives in coal mines will be found in "Revue Universelle des Mines," 2nd Series, vols. 18 and 19 (Liège, 1886). The British Report was published as an official document, "Final Report of H.M. Commissioners Appointed to Inquire into Accidents in Mines" (1886), and the French Report in "Annales des Mines," 1888, and in "Congrès International des Mines," 1889.

Flame

C. E. Bichel, "Testing Explosives," English translation by A. Larsen, London, 1905.

J.C.S.I., 1899, p. 7.

S.S., 1908, p. 408; 1909, pp. 323, 343.

P.S., 1910, p. 164.

Bulletin of the U.S. Bureau of Mines, No. 66.

Reproductions of flame photographs will also be found in A. Marshall, "Explosives," London, 1917.

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Galleries

The U.S.A. Gallery is described in detail in *Bulletin of the Bureau of Mines*, No. 15. This publication contains much interesting information on "Permissible Explosives," and together with *Bulletin* No. 66 should be studied by any one interested in American conditions. Unfortunately it does not give the composition of the explosives used.

The British Gallery at Rotherham is described with drawings in A.R., 1912, p. 83.

An account of the Continental galleries will be found in A. Marshall, "Explosives," and P. F. Chalons, "Les Explosifs Modernes."

The effect of turbulence on the ignition of gas mixtures is discussed in *Soc.*, 1919, p. 87.

Wrappers

"VIII. International Congress of Applied Chemistry," vol. iv. p. 138.

Size of Bore Hole and Gallery

S.S., 1910, pp. 221, 225; 1911, pp. 321, 344, 371.

Permitted Explosives

The composition of the explosives on the British Permitted List will be found in the following "Explosives in Coal Mines Orders":—

September 1st, 1913.
February 10th, 1914.
April 7th, 1914.
May 13th, 1914.
June 22nd, 1914.
August 29th, 1914.
January 15th, 1915.
January 28th, 1915.
April 1st, 1915.
July 3rd, 1915.
August 16th, 1915.
February 3rd, 1916.
April 14th, 1916.
April 26th, 1916.
September 21st, 1916.
November 21st, 1916.
May 9th, 1917.
November 5th, 1917.
May 30th, 1918.
August 2nd, 1918.
November 28th, 1918.
January 25th, 1919.

The composition of a large number of continental explosives is given in A. Marshall, "Explosives," London, 1917.

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Section VI.—PERCUSSION CAPS, DETONATORS AND FUZES

The actual mechanism of detonation is uncertain, but it is probably either a very rapid wave of compression raising the temperature locally in successive layers, or it is a vibration of the molecules of such a nature that it causes disruption into more stable forms of matter. In any case, when the majority of explosives burn the rate of burning increases until it reaches detonation, the velocity of detonation then remaining practically constant.

In order to be suitable for use as an initiator an explosive must be of such a nature that the acceleration is very rapid, burning passing into detonation in the least possible time. Further, it must keep well, and although for some purposes sensitiveness to shock is essential, this must not be so excessive that the manufacture and handling of the material is accompanied by undue danger.

No chemical compound fulfils all these conditions, and of the various ones that have been proposed from time to time only two have been found suitable for practical purposes, viz. mercury fulminate and lead azide. Of these mercury fulminate possesses fair acceleration, and is not unduly dangerous to handle when proper precautions are taken. It, however, is expensive, does not keep well, and the mercury fumes evolved during its explosion have a bad effect on brass cartridge cases. Also, like all mercury compounds, it is very poisonous, and consequently workers must be carefully protected from inhaling dust, and special means must be taken to dispose of effluent such as wash water, which may contain traces of mercury. This is particularly necessary when the factory is situated near rivers containing fish.

Lead azide, on the other hand, possesses excellent acceleration, and keeps well. It is less sensitive than mercury

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fulminate when in small crystals, but, unlike fulminate, it readily forms large crystals, and these are so sensitive that they explode when touched even when under water. It is somewhat less expensive and less poisonous than fulminate, but, on the other hand, the plant required for its manufacture is much more expensive.

Mercury fulminate is still the most used initiator, but lead azide is coming into more general use, and probably in the future will displace fulminate to a considerable extent. The properties required of an initiator depend a good deal on the conditions under which it is to be used. Blasting charges are always fired by means of a fuze or an electric current, so that, provided the initiator is sensitive to heat, there is no need for it to be sensitive to shock. On the other hand, except in the case of non-detonating explosives such as gunpowder, it is required to detonate the explosive and not merely to start combustion. Hence, detonators for blasting purposes require to be powerful, as otherwise the charge will either be merely ignited, or detonation will be incomplete.

Propellants, on the contrary, are not detonated, but ignited, so that a weak initiator is used, this firing a priming composition or "flaming mixture," which in turn fires the propellant. These flaming mixtures are of a very inflammable nature, and consist usually either of fine grain gunpowder or of antimony sulphide mixed with an oxidizing agent. In the case of small arms only slight power is available, that due to the fall of the hammer, so that an initiator very sensitive to shock must be used. With quick-firing guns more power is available. With heavy ordnance plenty of power is available, so that great sensitiveness is not required, and the initiator in this case usually contains no explosive properly so called, but is more akin to match composition. Naval guns and guns of position are frequently fired electrically, so that sensitiveness to heat only is called for.

Mercury Fulminate, $Hg(ONC)_2$.—This is an endothermic compound, the heat of formation being $-62{\cdot}9$ Cal. It is always manufactured by the action of ethyl alcohol on a solution of mercury in nitric acid. The reaction is a very

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violent one, and in order to obtain a satisfactory yield the manufacture must be carried out on what is practically a laboratory scale, only about 3 lbs. of fulminate being made at a time. Several recipes have been published differing only in minor details, but the following description gives a good general idea of how the manufacture is usually carried out.

The plant consists of glass bottles or retorts of about 50 litres capacity each, each being connected to a stoneware or silica worm condenser and with a brick scrubbing tower. Ample draught must be provided to draw off the fumes, as they are very poisonous. They consist largely of nitrous acid, aldehyde, ethyl nitrate, ethyl nitrite and unchanged alcohol, and are highly inflammable. The process is carried out by dissolving 500 grams of mercury in 4500 grams of nitric acid (D = 1400). The solution of mercuric nitrate thus obtained is warmed or cooled to 25° C., and 5 litres of 94 per cent. spirit added. The reaction starts after about 15 minutes, and lasts for about three-quarters of an hour. If, however, the temperature of the nitrate solution has been much below 25° C., it may be necessary to apply gentle heat in order to start the reaction, whereas if the temperature has exceeded 25 °C., the reaction may be uncontrollably violent. During the reaction the temperature rises to about 85° C., and much liquid distils over and condenses. This distillate is not of great value, but can be used to replace part of the spirit for the next batch. When the reaction is over the contents of the vessel are allowed to cool, about 1 litre of water is added, and the whole then poured out into a porcelain dish, and the fulminate thoroughly washed with cold water, first by decantation and then on a muslin filter. The wash waters are collected, made alkaline with lime, and the mercury thus precipitated, filtered off, and recovered. About 10 per cent. of the total mercury used is recovered by this means, and after the precipitated mercury has been filtered off the liquors can usually be safely run to waste, although, as a matter of safety, they should be dealt with so that they do not find their way by infiltration into any

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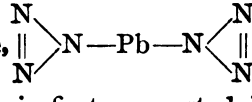
drinking water supply or into any stream stocked with fish.

The fulminate thus obtained is quite safe so long as it is preserved wet. It is only dried immediately before use, the drying being carried out by spreading it on paper trays in a stove heated to 35° C. These stoves usually take 100 lbs., and great care must be taken not to unload them until the fulminate is quite cold, fulminate being much more sensitive when warm. Thus obtained, it forms a grey powder, the yield being about 650 grams from 500 grams of mercury, which corresponds to about 90 per cent. of theory. If a little metallic copper and hydrochloric acid is added to the nitrate solution just before the spirit, an almost white product is obtained, but this is less pure than the grey.

Fulminate has a specific gravity of about 4·4, and 100 c.c. of water dissolves ·07 gram at 13° C., ·17 gram at 49° C., and ·8 gram at 100° C. It can be quantitatively estimated by titration with thiosulphate—

$$Hg(ONC)_2 + 2Na_2S_2O_3 + 2H_2O = Hg(S_2O_3)_2 + 2CN + 4NaOH.$$

Lead Azide,  . In making lead azide, metallic sodium is first converted into sodamide, $NaNH_2$, by heating it to 350° C. and then passing a brisk current of dry ammonia gas over the surface. Considerable heat is evolved, and by using a fairly brisk stream of ammonia the temperature is maintained during the reaction without external heating. The reaction is easily followed by testing the gas at the exit, a rapid falling off in the hydrogen content indicating that the whole of the sodium has been used up. The sodamide thus formed is next converted into sodium azide by heating it to 150°–250° C. in a stream of nitrous oxide (laughing gas)—

$$2NaNH_2 + N_2O = NaN_3 + NaOH + NH_3$$

The sodium azide thus formed is dissolved in water, and lead salt thrown down by double decomposition with lead

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acetate. It forms a white crystalline powder which is almost insoluble in cold water, and not much more so in hot water. It has a tendency to form large crystals, and these may explode spontaneously even when under water. Small or moderate crystals are less sensitive to shock and heat than mercury fulminate, and owing to its high acceleration it is more effective as an initiator. Thus it has been found that whereas ·25 gram of fulminate was required to detonate trinitrotoluol, ·05 gram of lead azide was sufficient under the same conditions. It is chiefly used in conjunction with tetryl or T.N.T. in composite detonators.

Other sensitive chemical compounds, such as diazobenzene nitrate, nitrodiazobenzene nitrate, lead picrate and nitrogen sulphide have been proposed for use as initiators from time to time, but with no success, as in all cases the acceleration is too slow.

Caps.—Cap composition for small arms is usually a mixture of fulminate of mercury, potassium chlorate, antimony sulphide and other oxidizable matter to which ground glass is frequently added to increase the sensitiveness. For ordnance, where more power is available, the fulminate is usually omitted. Sometimes gelatine or gum arabic is added to give coherence to the composition, but the addition is by no means necessary unless the mixing is done wet.

The following table shows the composition of some British and Austrian caps:—

	British S.M. (Cordite).	British (Cordite).	British Q.F. (Cordite).	Austrian, for rifles.	Austrian, for shot guns.	Austrian, for friction tubes.
Fulminate ..	8	19	—	13·7	33·9	—
$KClO_3$ ..	14	33·3	12	41·5	21·6	66·2
Sb_2S_3 ..	18	42·9	18	33·4	—	33·1
Meal G.P. ..	1	2·4	1	—	—	—
S.	1	2·4	1	—	—	—
Glass ..	—	—	1	10·7	43·2	—
Gum ..	—	—	—	—	—	·7
Gelatine ..	—	—	—	·7	1·3	—

Herz has suggested the use of copper ammonium thiosulphate, prepared by treating copper ammonium sulphate

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with sodium thiosulphate, and lead thiosulphate in conjunction with potassium chlorate, and has claimed good results with the following mixtures, although they do not seem to have come into use:—

$KClO_3$	57·3	54·5	51·5	53
$Cu(NH_3)_4S_2O_3$	42·7	40·5	37·1	27·5
PbS_2O_3	—	—	7·4	5·5
Sb_2S_3	—	—	—	11·0
Glass	—	4·0	4·0	3·0

In preparing cap composition the powdered ingredients are sieved separately and then mixed, either in the wet or in the dry state. In Great Britain and in France the mixing is usually done dry, but in Austria and Germany wet mixing is frequently preferred. When the mixing is carried out in the wet way it is absolutely necessary to add gum or other binding material to prevent the ingredients separating. Sufficient water, in which the binding material has been dissolved, must be added to form a thick mud, but an excessive amount must be avoided, as if present it will carry away some of the chlorate and also interfere with the granulating of the material. Too little water, on the other hand, renders the composition dangerous. In any case, great care must be taken that none of the composition gets on to the edge of the mixing dish and becomes dry. In order to avoid loss of chlorate by solution in the water, alcohol is sometimes added, as by this means the solubility of the chlorate is greatly lowered. In any case, after mixing the composition is granulated by rubbing through horse-hair or silk sieves, and then dried and sifted. It is not advisable to load the caps with the wet composition and then dry them as was done at one time, as under these conditions the composition is apt to form a hard mass which is unduly sensitive. For use in the wet process the fulminate is not, of course, dried, the paste containing about 15 per cent. of water being used. When mixing by the dry method the ingredients, with the exception of the fulminate, are first mixed, the fulminate then added and the mixing repeated. The object of this

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procedure is, of course, to do as much of the mixing as possible before adding the dangerous fulminate. Owing to the dangerous nature of the substances dealt with the mixing is done in very small quantities, from one-half pound to two pounds being the usual charge. The plant used is very simple, and consists either of a papier maché drum revolved by hand, or of a cloth or light leather "jelly-bag." When the drum method is used the preliminary mixing of the ingredients other than the fulminate is assisted by the addition of soft rubber balls, thus converting the papier maché drum into a sort of crude ball mill. These balls are removed, however, before the fulminate is added.

The "jelly-bag" is probably the best and most used mixing machine, and is shown in Fig. 22. It consists of a conical bag of cloth or light leather, the upper and wider end being attached to a frame. To the inside of the apex is attached a cord carried over pulleys to the operator, who is at a safe distance and behind a suitable screen. By pulling the cord the bottom of the bag is raised, thus turning over the contents and mixing them, a movable stop being provided to prevent the cord being pulled too far and thus causing the contents of the bag to be spilt. On releasing the cord the bag is returned to its normal position, either by the weight of the composition or by a counterpoise. Sometimes rubber discs are added to make the mixing more thorough. During the mixing a vessel of water is placed under the bag to catch any composition which may be accidentally upset, but when mixing is complete this is replaced by a paper box. The operator then retires behind the screen, removes the movable stop, and, by pulling the cord, turns the bag outside in, the composition being guided into the cardboard box by the conical funnel surrounding the bag. The method is extremely safe, as even should a charge explode during mixing the operator is well protected. To allow the operator to watch the bag a narrow slit may be made in the screen, but a window of Triplex safety glass would probably be more satisfactory. This has been tested officially with most encouraging results,

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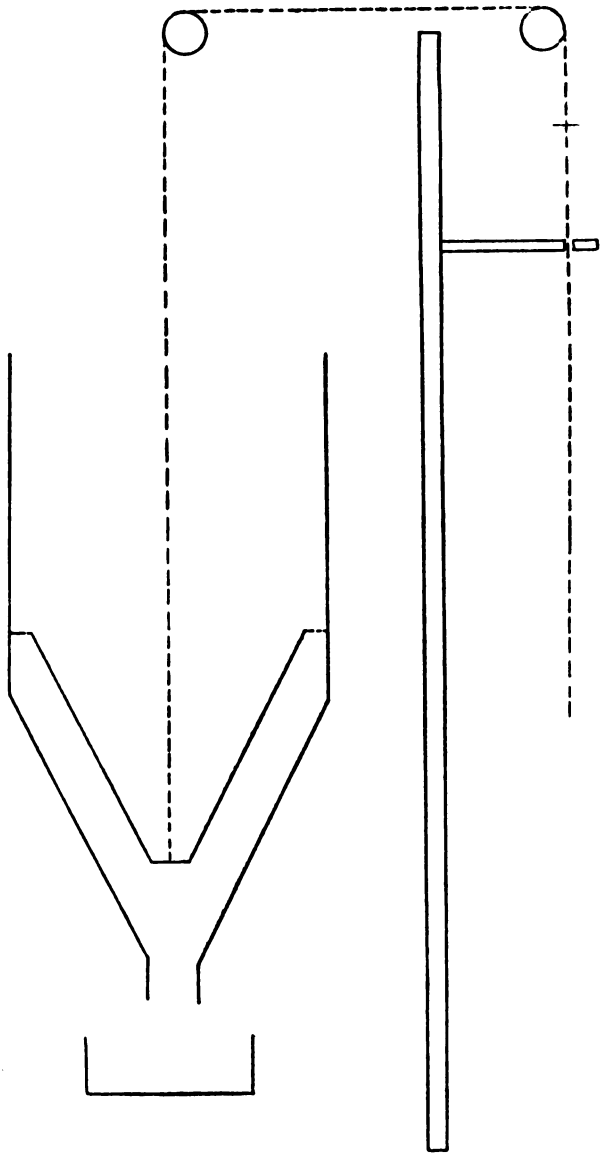

FIG. 22.—Jelly-bag Mixing Machine for Cap and Detonator Composition.

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the following description of the test being taken from *A.R.*, 1917 :—

"A block of Triplex Safety Glass 2½ in. thick was securely fixed by ½-in. angle iron plates to a screen of timber 3 in. thick, and 4500 grains weight of fulminate of mercury was fired close underneath the glass. The result was to shatter the wooden screen work, large pieces of timber flying 150–200 ft. in the air, but the Triplex glass, although cracked in all directions, was found to be absolutely whole—a clear demonstration of the great protection to an operator that would be afforded by this glass during work with detonators."

The metallic cap cases are stamped out of sheet copper,

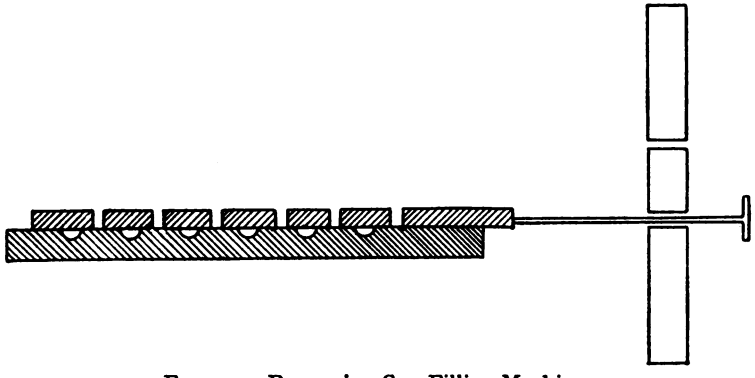

FIG. 23.—Percussion Cap Filling Machine.

or, less frequently, brass, and are varnished internally. The composition is filled in in the following manner: The cases are placed in depressions in a bronze plate, called a "hand," 1000 being the usual number. Another plate, usually made of ebonite, is then placed on the "hand," this plate having perforations corresponding to the depressions in the "hand," but is placed in such a position that the rows of perforations come between the rows of depressions (Fig. 23). The thickness of the upper plate is so chosen that the correct weight of composition just fills the perforations. Rather more than the weight of composition required to fill 1000 caps is then placed on the upper plate and brushed into

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the perforations so as to fill them, any excess of composition being removed with a soft brush. The operator then retires behind a screen and slides the upper plate along the lower until the perforations correspond with the depressions, and the composition falls into the caps. The upper plate is then removed, and the "hand" placed in a hydraulic or mechanical press provided with a number of pistons, each piston entering one cap. The pressure applied is about 200–250 lbs. per cap, and usually there are sufficient pistons in each press to press one or two rows of caps at a time, the "hand" being moved forward in order to allow the next row to be treated. After pressing, any loose composition is fanned away and the caps then varnished by placing a single drop of alcoholic shellac on the top of the composition, after which the alcohol is dried off at a low temperature. If the caps are to be transported a tin disc must be inserted into each (see page 14), and this is frequently done in any case. It is fixed by pressing in presses similar to those used for the composition, and is subsequently varnished.

Caps only contain about ·5–·6 grain (say, ·04 gram) of composition, so that the amount of composition required for a "hand" holding 1000 caps does not exceed 50 grams. Consequently it is not difficult to provide adequate protection in the way of shields for the workers. Undoubtedly Triplex Safety Glass windows in these shields will prove a great convenience by allowing the operations in progress to be watched, while at the same time providing adequate protection.

Officially, caps are defined as containing not more than half a grain of composition, or not more than ·6 grain if it contains less than 25 per cent. of fulminate. If they contain more they must be treated as detonators (see page 14). Caps can be roughly tested by observing the marks produced when they are flashed against a sheet of white paper at a given distance, or, more accurately, by a modification of the crusher gauge. More elaborate methods determine the length or duration of the flame, and the temperature of explosion is determined by an electrical or optical

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pyrometer. References to these methods will be found at the end of the section.

The use of mercury and antimony is objectionable with brass cartridge cases, as these metals have a bad effect on the brass, but so far no satisfactory substitutes seem to have been discovered. In the same way, the chloride resulting from the chlorate causes rusting. An all-organic initiator would be a valuable discovery, but so far has not been attained.

Detonators.—Detonators being required to produce detonation and not ignition require to be much more powerful than caps. Until comparatively recently they have been chiefly composed of a mixture of 80 per cent. of fulminate of mercury and 20 per cent. of potassium chlorate rammed into copper tubes, although a great many French detonators contain only fulminate. In recent years composite detonators have been largely used in which T.N.T. or, better, tetryl is first pressed into the case and a detonating composition of fulminate and chlorate or of lead azide pressed on to the top of this. These detonators are practically a detonator and primer combined, the fulminate or azide detonating the T.N.T., or tetryl, which in turn detonates the cartridge. They have the advantage of being cheaper than non-composite detonators, and of involving the use of much less of the dangerous fulminate. The small amount of fulminate used, however, has one disadvantage, and that is that the effect of deterioration is much more marked. Fulminate does not keep well, and if the amount is reduced to a minimum, as is the case in composite detonators, there is much greater chance of irregular results being obtained. This objection does not, of course, apply to composite detonators made up with lead azide in place of fulminate.

The size of detonators has been standardized in all countries, and the following table shows the sizes, together with the charge of fulminate-chlorate mixture (80 per cent. fulminate), and the charges for a few composite detonators of equal strength :—

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No.	External dimensions.		Charge (grams).		
	Length.	Diameter.	Fulminate—$KClO_3$.	T.N.T. or Tetryl.	Fulminate.
	mm.	mm.	·3	—	—
1	16	5·5	·4	—	—
2	22	5·5	·54	—	—
3	26	5·5	·65	—	—
4	28	6·0	·8	—	—
5	30–32	6·0	1·0	·3	·3
6	35	6·0	1·5	·4	·4
7	40–45	6·0	2·0	·75	·5
8	50–55	6·7	2·5	·9	·5

Of these Nos. 5 and 6 are usually used for nitroglycerine explosives, and No. 7 for ammonium nitrate explosives. No. 8 is not much used, although probably better results would be obtained by employing somewhat stronger detonators than is the custom.

French composite detonators have been manufactured containing picric acid, but they are somewhat uncertain, and, in any case, picric acid in contact with copper is most objectionable. The copper tubes for detonators are stamped out of sheet copper, and on account of the dimensions only the very best soft copper can be used. The methods employed in the filling operation are much the same as those used in filling caps. The empty cases are filled into perforations in an ebonite plate, each perforation being lined with brass to prevent distortion, and being of such a size, that the detonator is a snug but not a tight fit. Usually only 100 detonators are treated at a time, the charge for each being measured out in the perforations of a sliding plate in exactly the same way as when caps are being filled. The pressing is carried out in presses with 100 pistons, each piston being a loose fit in the detonator so as to avoid friction. The pressure employed is about 250 kg. per cm.^{2} (60 kg. per detonator), and this raises the density of the charge to about 2·2. By using higher pressure higher density is attained, but the effectiveness of the detonator falls off considerably. After pressing they are rumbled with sawdust to remove any loose composition, and then packed.

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The British regulations for packing specify that the detonators shall be packed in sawdust in tin boxes lined with felt or paper, and that the ends shall rest against a felt pad. These tin boxes are packed in outer cases, and if the package thus formed contains over 1000 detonators the outer case must be double, with a space of not less than 3 in. between the walls. If over 5000 detonators are in one case, the case must be provided with handles for lifting, and the maximum number of detonators that can be packed in any case is 10,000.

Detonators in which the one end is left open are fired by means of a safety fuze, the end of the fuse being pushed down until it touches the composition and then held in place by constricting the mouth of the detonator with a pair of pliers. Electrical firing, however, is rapidly increasing, and detonators for this purpose are known as electric detonators, and are of two types, viz. high tension and low tension. In the high-tension detonators firing is brought about by an electric spark passing between two points and igniting a flaming mixture. They are not much used as, under working conditions, the high-tension current needed is liable to short circuit, and the detonators cannot be tested before use. Low-tension detonators are actuated by the electrical heating of a fine platinum wire, only a low voltage current (2–3 volts) being required. They can be tested before use by determining their resistance on a Wheatstone bridge, a sensitive galvanometer and a very small current being used. The platinum wire might actually be embedded in the composition, but this system would be unreliable, as it would probably be broken during pressing, and, in any case, there would be danger of friction being set up through movements of the free ends. The system always employed is to solder a very fine platinum wire on to the leads which are in turn cemented into the open mouth of the detonator by means of sulphur or bitumen. Previous to this, however, a bead of a flaming mixture of sulphide of antimony and chlorate of potash is melted on to the platinum wire. In use, the heating of the platinum wire ignites this flaming

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mixture, which in turn ignites the detonator composition. As a source of current a small dynamo is generally used, the armature being revolved by depressing a plunger, this being geared on to the armature by a rack and spur-wheel. When almost at the bottom of its stroke this plunger closes the circuit with the detonator, this arrangement being provided so that no current passes until the speed of the armature, and hence the voltage, has had time to reach a sufficient magnitude to make ignition certain.

Several methods have been proposed for testing detonators, such as the lead block test, in which the detonator is fired in a lead block much the same as the Trauzl block (see Section VIII.) only smaller; the crater test, in which the detonators are fired in contact with a plate of $\frac{1}{2}$-in. lead and the craters produced compared; and the nail test. This latter is the best, and is carried out as follows: The detonator is attached to a 4-in. wire nail by means of a copper wire round its centre, but is prevented from actually touching the nail by two bands of copper wire placed round the detonator near each end, the wire being ·025 in. diameter (23 B.W.G.). The bottom of the detonator points towards the head of the nail, but is $1\frac{3}{4}$ in. from it. The whole is then suspended, nail upwards, by the leads, as shown in Fig. 24, and then fired, care being taken that the nail cannot be hurled against any hard substance. The angle of deformation of the nail is taken as a measure of the strength of the detonator.

Safety Fuze.—The object of safety fuze is to ignite the detonator at an interval of time after the fuze has been lighted, thus allowing the shot firer to get to a safe distance. A fuze must be reliable, and not be apt to go out; it must be capable of burning under water when subaqueous blasting is being carried out; and must burn at a constant rate so that the explosion of the charge does not take place before the firer has attained a place of safety. All safety fuzes consist of a core of fine grain gunpowder enclosed in an outer case of jute, the outer case being more or less waterproofed according to the type of fuze desired. To prevent the case smouldering the jute is frequently impregnated with

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ammonium phosphate, or other suitable neutral salt. Modern safety fuze has a very steady rate of burning of about 2 ft. per minute.

The plant necessary for "spinning" fuze is shown diagrammatically in Fig. 25, and consists of a hopper containing gunpowder, the lower end of which terminates in a fine nozzle. A frame containing from seven to ten reels of jute yarn revolves round the hopper, the yarn being carried down

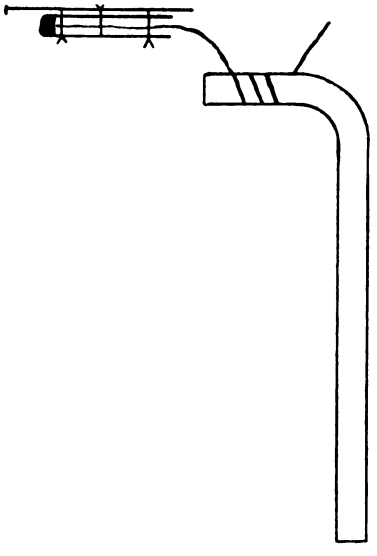

FIG. 24.—Testing Detonators.

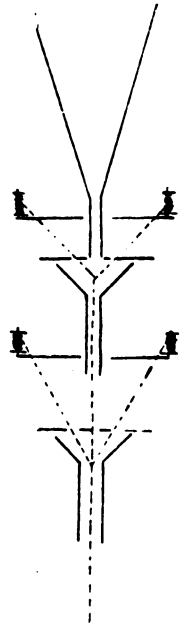

FIG. 25.—Fuze Spinning.

close to the nozzle. As the powder flows out of the nozzle the revolving reels enclose it in a loose spiral of thread which then passes through a narrow tube. As it issues from the lower end of this tube another set of reels revolving in the opposite direction spins another spiral of thread round it, after which the fuze is wound on a drum and is ready for waterproofing, etc. In order to maintain an even flow of powder from the hopper a thread is sometimes made to pass through it, the motion of this thread preventing the

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orfice becoming choked, and at the same time assuring a more equal flow of powder.

For ordinary use in damp situations sufficient water-proofing is attained by passing the fuze through tar, after which it is wound with tape, the turns overlapping, and then again tarred. For subaqueous work, however, the fuze is usually first treated with a priming coat composed of Stockholm tar and low grade gutta-percha, and then coated with pure gutta-percha. To preserve this from mechanical damage it is usually wound with tarred yarn.

Instantaneous Fuze.—This is a rapidly burning fuze intended for firing several shots simultaneously. It consists of a wick impregnated with meal powder and then loosely spun over with yarn. The rate of burning is about 300 ft. per second, but it is little used now, having been replaced by electric detonators or detonating fuze.

Quick-Match.—This is much the same as instantaneous fuze, but is unwound so that its rate of burning is much slower, viz. about 15 ft. per minute. It is used to some extent in fireworks, *e.g.* for firing the bursting charge in the heads of rockets.

Slow-Match.—This is a loosely woven hemp cord slightly impregnated with potassium nitrate, so that it smoulders slowly. It is used chiefly in conjunction with pyrophoric alloys for cigarette lighters, and takes the place of the tinder used with the old-fashioned "flint and steel." It is also used to some extent for delayed action mines in warfare.

Detonating Fuze.—This is practically a very narrow cartridge of some detonating explosive in a metal case, T.N.T. or tetryl being the explosive most used. It is usually about 4 mm. in diameter, and is put up in coils of 50 ft. or more. A guncotton detonating fuze was at one time made in France by filling dry powdered guncotton in a lead pipe 15·5 mm. external diameter and 12 mm. internal diameter. The ends of the pipe were then closed and the whole drawn out by passing through a series of holes in a draw-plate in much the same way that wire is drawn. These holes diminished in diameter from 15·5 to 5 mm. by intervals of

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·5 mm., and then from 5 mm. to 4 mm. by intervals of ·2 mm. This fuze was said to give satisfactory results, but never came into general use, and has now been displaced by fuzes containing T.N.T. These are made by sucking molten T.N.T. into a lead pipe by means of a vacuum, the pipe being kept hot in a water-bath. After cooling, the fuze is drawn out through draw-plates, but as much narrower tubes can be filled by sucking in a liquid than is the case when they are loaded with a solid, like guncotton, the same number of drawings is not necessary. The rate of detonation is very constant, about 5000 metres per second, and the fuze is much used for determining the rate of detonation of explosives (Section VIII.). It is also used for the simultaneous firing of several shots, and in warfare attempts have been made to employ it for the destruction of wire entanglements. It is fired by means of a detonator. It should be noted that *pure* T.N.T. must be used for the manufacture of fuze, as the crude product is apt to become plastic on cooling, and will then not detonate properly.

A somewhat different type of detonating fuze is used in the Austrian army. This consists of threads impregnated with mercury fulminate phlegmatized by the addition of 20 per cent. of paraffin wax. When fired with a suitable detonator its rate of detonation is about 6000 metres per second, but if ignited it burns quietly, unless it becomes hot enough for the paraffin to meet and run away from the fulminate.

Shell Fuzes.—These are of two types, viz. time fuzes and percussion fuzes, the former being intended to explode the shell in the air after it has travelled a certain distance, and the latter to explode it on striking. Shells with time fuzes are frequently also fitted with percussion fuzes, so that they explode on striking should the time fuze fail, whereas percussion fuzes are often connected with a short time fuze so that explosion only takes place a short time after the shell has struck, thus giving it time to penetrate. This type of fuze, delayed action fuze, is much used for armour piercing shell, and for the bombardment of deep dugouts, as an

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ordinary percussion fuze would explode the shell on the surface, and thus do comparatively little damage.

Time fuzes consist of one or more rings of slow-burning gunpowder situated in the nose of the shell, and communicating with the burster charge. The fuze is fired by a simple cap actuated by the shock of the propellant. The rings are movable, and by rotating them the length of the powder train between the firing cap and the burster charge can be altered. The rings are graduated with a scale showing the number of yards of travel of the shell, and the fuze is set for the given range before the shell is inserted in the gun.

Percussion fuzes are set either in the nose or in the base of the shell, and are actuated by the sudden retardation when the shell strikes, driving a pin on to a cap. To prevent the shock of the discharge actuating the pin, special safety arrangements are provided. These may take several forms, *e.g.* the pin may be held back by a powerful spring, the resistance of which can only be overcome by a very violent blow on the nose of the shell, or it can be provided with a safety catch which is only released by the centrifugal force set up by the rotation of the shell. As stated above, the percussion fuze is often made to fire a short time fuze in order to allow the projectile to penetrate before bursting.

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Percussion Caps

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The following references are the methods of testing caps: *J.S.C.I.*, 1905, p. 381; 1906, p. 241.

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Detonators

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P.S., xii. p. 134.

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A very interesting critical examination of the various methods of testing detonators is given in *Bulletin of the American Bureau of Mines*, No. 59. In this publication the methods are described in detail.

Safety Fuze

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Detonating Fuze

S.S., 1907, p. 173; 1910, p. 169; 1913, p. 312.
See also Chalons, " Les Explosifs Modernes," Paris, 1911, p. 424.

Shell Fuzes

These are fully illustrated and described in the Official Publications, " Treatise on Ammunition " and " Handbook on Ammunition."

Also in R. Willie, " Mechanische Zeitzünder," Berlin, 1911. D. T. Hamilton, " Shrapnel Shell Manufacture," New York, 1915.

Accidents

Accounts of recent accidents that have occurred in Great Britain in connection with the manufacture of fulminate, detonators, etc., will be found in *S.R.*, 186, 188, 196, 199.

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Section VII.—MATCHES, PYROPHORIC ALLOYS AND PYROTECHNY

Matches

Until 1805 the only mechanical means of obtaining fire as distinguished from the use of the burning glass, consisted in striking sparks with flint and steel, and by this means igniting tinder. Curiously enough during recent years this method has again come to the front in a modified form, the flint being replaced by special pyrophoric alloys, and the tinder by slow-match or by a wick soaked in petrol. In 1805 Chancel introduced splints of wood tipped with a mixture of sugar and potassium chlorate mixed with gum, these being ignited on a pad of asbestos moistened with concentrated sulphuric acid, and in spite of their obvious inconveniences these matches remained in use as late as 1844. The first friction match was made about 1806, and contained phosphorus, but was not a practical success, and it was not until 1827 that a satisfactory friction match was produced. These were tipped with a mixture of potassium chlorate and antimony sulphide, and were ignited by drawing them sharply through a piece of glass paper held between the finger and thumb.

The modern match industry is a large one, 17,250,000 gross boxes, each containing on the average 60 matches, being manufactured in Great Britain in 1910, and this figure only representing about half the consumption, a similar amount being imported. In the United States the consumption is about 250,000,000,000 matches per annum, corresponding to about nine matches per day per head of population, a figure corresponding closely with the consumption per capitum in this country.

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Broadly, matches can be divided into two divisions, viz. non-safety, or strike-anywhere matches, and safety matches which are supposed to ignite only when rubbed on a prepared surface, but which, as a matter of fact, can usually be ignited by drawing them smartly across any smooth surface of low heat conductivity, such as a sheet of glass or paper.

Composition for match heads is made up of five parts, viz. (1) oxidizable matter, (2) oxidizing agents, (3) inert gritty matter (to increase friction), (4) colouring matter, and (5) binding material. Striking surface for safety matches is usually composed of inflammable matter only, with or without the addition of grit to increase friction.

As oxidizable matter, yellow phosphorus was originally used, but owing to its poisonous properties causing necrosis (" phosy-jaw ") among the workers its use is now prohibited in almost all countries. It has been replaced by sulphide of phosphorus, sulphide of antimony, zinc sulphide and scarlet phosphorus, or a mixture of two or more of these. Sulphide of phosphorus, P_4S_3, is generally used for strike-anywhere matches, although a very small proportion of this type of match is made up with scarlet phosphorus, and the use of lead, tin and copper thiocyanate has been proposed. Sulphide of antimony, Sb_2S_3, is almost universally used for safety matches.

As an oxidizing agent potassium chlorate enters into the composition of almost all match heads, although potassium bichromate, manganese dioxide and lead dioxide are also used.

The inert gritty matter generally takes the form of powdered glass, whereas pigments, such as ochre, ultramarine, etc., are used to improve the appearance. The binding material is added in order to make the composition adhere to the splint or taper, and is usually gum arabic or dextrine.

As a striking surface, antimony sulphide and red phosphorus are usually made up with gum arabic or dextrine, and then painted on to the sides of the box.

In the case of wax matches the flame from the head is

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sufficient to ignite the taper, but with wooden matches the splint must be tipped with some inflammable material in order to convey the ignition from the head to the wood. This is usually done by dipping the splint into molten paraffin wax before putting on the head. In France, and to a lesser extent in America, however, sulphur is frequently used for this purpose. Such matches burn at first with a blue flame, evolving sulphur dioxide and giving practically no light, and have therefore been named "wait-a-minute matches."

To prevent the splints glowing after the flame has been extinguished most modern wooden matches are impregnated with certain inorganic salts. For this purpose sodium tungstate is one of the most effective salts, but it is somewhat expensive, so that sodium or ammonium phosphate is generally used, although zinc sulphate, magnesium sulphate, alum and phosphoric acid have also been employed.

Various kinds of timber are used for the splints of wooden matches, such as pine, aspen, spruce, etc., and the same applies to the wooden boxes in which the matches are put up.

In making up match composition the ingredients are ground separately in the dry state, and then mixed wet. As a rule the oxidizable matter and part of the inert matter are mixed together with part of the gum solution and, separately, the oxidizing agent is mixed with the rest of the inert matter and binding solution. The two mixtures thus obtained are then mixed together to form the paste ready for use. By adopting this procedure danger of fire is reduced to a minimum, as the oxidizable matter and oxidizing agent are not brought together until both are thoroughly wet.

The actual tipping of the matches is a purely mechanical operation, and great ingenuity has been shown in designing machines for carrying out the operation with the minimum amount of labour. These machines cannot be described in detail in a book of this nature, but the following description will give a general idea of the principle on which they act.

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When making square matches the splints are usually cut out in a separate machine, but in the case of round or grooved matches the splints are usually cut out by the same machine that tips them. The taper for wax matches is, of course, made separately and fed into the match-making machine after being cut to the correct length. Otherwise the operation of making wax matches is similar to that used for wooden matches, except that, of course, they are neither impregnated nor tipped with wax. In the match machine the splints are fed from a hopper into grooves in the iron match plates, the splints being made to enter the grooves end on. These plates are attached to an endless chain, and the hopper is so arranged that just after the splints have been wedged into the plates the chain passes over a pulley, thus leaving the matches with the free ends pointing downwards. The plates carrying the splints next pass over a bath of the impregnating solution so that the splints are immersed, and then over a bath of molten paraffin so that the free ends dip into the wax. This wax usually has a melting point of 39° C., but is maintained at a temperature of 100°–105° C. by means of a steam jacket. After receiving their coating of wax the ends of the splints pass through a third bath containing the match-head composition. This is made of a suitable consistency, so that a fairly large "blob" of it adheres to the end of the splint, and this constitutes the head. After passing this bath the chain is carried upwards to the ceiling of the building, or, what is better, through the ceiling into an upper chamber. Here it passes over a long series of rollers in order to allow the head to dry, the drying operation being assisted by hot air. The chain then returns to a point near the hopper, where the finished matches are unloaded from the plates, and mechanically packed into the boxes, these boxes being then mechanically made up into the familiar packets of one dozen boxes. These packets are subsequently made up by hand into packets of one gross.

Modern match machines are of considerable size, the endless chain being as much as 700 ft. long, and taking an hour to make one circuit. Such a machine will turn out

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about 1000 gross boxes of matches in a single day, corresponding to nearly 9,000,000 matches, and only requires eight girls to look after it.

As regards the composition of match-head material, various recipes are in use. The heads of the old white phosphorus non-safety matches were composed of—

White phosphorus	2·5 kg.	·2 kg.
Lead dioxide	—	24 kg.
Ferric oxide	·5 kg.	—
Glass	2·0 kg.	—
Glue	2·0 kg.	—
Dextrine	—	6 kg.
Water	4·5 litres	4 litres

but the use of white phosphorus is now prohibited, modern strike-anywhere matches being made with sulphide of phosphorus, or, to a very minor extent, with scarlet phosphorus. Examples of such compositions are—

P_4S_3	6	—
Scarlet Phosphorus ..	—	10
$KClO_3$	24	45
ZnS	6	—
Ochre	6	—
$CaCO_3$	—	2
$CaSO_4$	—	5
Glass	6	22
Glue	18	10
H_2O	34	45

Of these the former is a French recipe, 1 kg. of the wet composition being sufficient for about 100,000 matches. Unfortunately all matches containing sulphide of phosphorus are apt to deteriorate in moist air with the liberation of sulphuretted hydrogen. This can be remedied to some extent by varnishing the head with shellac or collodion, but several proposals have been made for non-safety match-head composition in which no sulphide of phosphorus is used. The use of scarlet phosphorus, made by boiling white phosphorus with phosphorus tribromide, is mentioned above, but has never come into general use, although matches made with

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it are on the market. In Germany thiophosphites, made by heating a metallic sulphide such as ZnS, Sb_2S_3, or Cu_2S with red phosphorus in an atmosphere of carbon dioxide to 450° C., have been used, and good results are claimed with the following mixture :—

Zinc thiophosphite	·30
$KClO_3$	60
ZnO	5
$CaSO_4$	3
$CaCO_3$	5
Glass	10

Barium cupro thiosulphate, $BaCuS_4O_6$, obtained by precipitating two molecules of sodium thiosulphate with one molecule of cupric chloride and one molecule of barium chloride, has also been employed, as by its use a strike-anywhere match containing no phosphorus at all can be prepared. The following composition is recommended :—

$BaCuS_4O_6$	13
$KClO_3$	58
$CaSO_4$	10
S	3·7
Iron filings	4·3
Gelatine	11

Safety matches, as a rule, are made up with sulphide of antimony, and are ignited by rubbing on a surface of sulphide of antimony and red phosphorus. Numerous recipes for composition are in use, but the following may be taken as typical :—

Head.		Striking surface.	
Sb_2S_3 ..	24	Red Phosphorus	1
$KClO_3$ ..	32	Sb_2S_3	·25
$K_2Cr_2O_7$ ..	12	Carbon	·50
PbO_2 ..	24	Dextrine ..	·30
Glass ..	2		
Gum arabic	4		

but the compositions used by different makers vary widely, and sulphur, manganese dioxide and ferric oxide are frequently added.

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Pyrophoric Alloys

One of the oldest methods of obtaining fire was by striking a spark from flint and steel and using this to ignite some inflammable material, such as tinder. The introduction of matches led to the abandonment of this tiresome and clumsy process, but the discovery of pyrophoric alloys of cerium in 1903 led to its revival, and the modern "flint and steel" or "briquet," to use the French term, of which there seems to be no translation, became such a serious rival to matches that heavy taxes were imposed on them by those countries in which the match industry was a State monopoly, or in which matches were taxed for revenue purposes. The outbreak of war gave a further impetus to the pyrophoric alloy industry, as the "briquet" was preferred to matches by the troops owing to its lessened sensibility to damp and to the fact that the small quantity of petrol required could always be obtained from the mechanical transport. The "briquet" also became increasingly popular with the civilian population owing to the scarcity of matches.

Opinions differ as to the reason for the pyrophoric nature of cerium alloys. The pure metals of the cerium group are only slightly pyrophoric, although their temperature of ignition is low, and it has been suggested that the pyrophoric nature of the alloys is due to the increased hardness allowing finely divided dust to be projected into the atmosphere and ignited partly by the heat generated by the friction and partly by spontaneous combustion due to the large surface exposed to the oxygen of the air. It seems more probable, however, that the pyrophoric effect is due to the formation of highly inflammable suboxides.

For the preparation of pyrophoric alloys it is not necessary to use pure cerium, it being customary to make use of the crude mixture of rare earths left over as a waste product when the thorium is extracted from monazite sand for making incandescent gas mantles. In order to reduce this to the metallic state various methods have been proposed, such as reduction with metallic calcium, but industrially

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the process is always carried out electrically. This can be done by the electrolysis of the oxide in fuzed cerium fluoride, and on the laboratory scale this procedure gives excellent results. Unfortunately the melting-point of the fluoride is very high, about 1000° C., so that it has not been found possible to use this process on an industrial scale, and for manufacturing purposes it is usual to electrolyze a mixture of cerium chloride and chlorides of the alkali earth metals. In order to obtain satisfactory results the cerium chloride must be dry and free from oxide and oxychloride. As the chloride is soluble in alcohol, whereas the oxide and oxychloride are insoluble, separation can be effected by use of this solvent, but it is found better to obtain the pure, dry chloride by heating the crude chloride with sal-ammoniac.

The electrolysis is carried out in a graphite crucible with an iron cathode, and considerable difficulty is met with owing to the tendency of the metal to remain distributed in the electrolyte in very finely divided particles, or in the colloidal form.

Various proposals have been made for suitable electrolytes, some investigators recommending a mixture of two molecules of cerium chloride and one molecule of calcium chloride, while others claim good results with mixtures of calcium chloride and fluoride and barium fluoride. For example, it is stated that excellent results are obtained by fuzing three parts of barium chloride and three parts of calcium fluoride with eight parts of calcium chloride in an electric furnace, and then adding ten parts of cerium fluoride.

The metal obtained by electrolysis is usually cast into blocks weighing from two to twenty pounds, and contains about 80 per cent. of cerium, the balance being chiefly other rare earth metals, and in this state it is quite unsuited for pyrophoric purposes. A satisfactory pyrophoric alloy must be sufficiently hard to allow sparks to be struck with ease without being too hard, and at the same time it must be sufficiently tough not to crumble away. In order to obtain these properties it is usual to alloy the crude cerium metals with about 30 per cent. of iron, a little zinc being

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sometimes added to convey hardness, and sometimes a little copper to make the alloy tougher. The alloying is carried out by heating iron powder in a graphite crucible under a layer of an easily fuzible salt. The cerium is then added little by little when it melts and slowly dissolves the iron. The fuzible salt is used, of course, to protect the alloy from the action of the air, but in spite of this considerable loss takes place through oxidation, so that a pound of cerium does not give much more than a pound of alloy. When the whole of the iron has been dissolved the melt is cast either into a single block, which is afterwards cut up for sale, or into thin rods. Auer metal No. 2 is made somewhat differently, as the alloy is cast into a block and then ground to a powder, and this then moulded and heated until it sinters together. The object of this process is to obtain a finished stick that is impregnated with the lower oxides, and improved pyrophoric properties are claimed to be obtained by this process.

PYROTECHNY

The art of making fireworks is very ancient, the first use of explosives being for pyrotechnic purposes. The term "fireworks" includes two classes of goods, viz. (1) appliances for producing illumination, either for display purposes, *e.g.* Bengal fire, display rockets, Roman candles, etc., or for military or life-saving purposes, *e.g.* "star" shell, Holme's buoys, etc., or for purposes of signalling, *e.g.* Verey stars, marine rockets; and (2) appliances for producing sound either for display, *e.g.* crackers, or for signalling, *e.g.* fog signals, maroons, etc. The explosive or combustible materials used in firework construction can be divided into three broad groups, viz. slow burning mixtures for producing illuminating effects, such as golden rain, coloured fire, stars, etc., more rapidly burning mixtures used as propellants, *e.g.* for rockets, catherine wheels, etc., and rapidly burning mixtures used for bursting charges for rockets and for the production of sound. In some cases

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guncotton us used as a sound producer, but otherwise all the combustible material, both fast and slow, used in pyrotechny is of the gunpowder type, additions of metal filings being made in order to produce sparks and barium, copper, strontium, etc., salts being added in order to produce colour effects. For display purposes chlorates are largely used, as they give more brilliant effects than nitrates, probably largely owing to the greater volatility of the chloride produced during combustion, but the use of chlorates in the presence of sulphur is prohibited in Great Britain. The danger of chlorate-sulphur mixtures is largely due to the fact that some of the sulphur becomes oxidized to sulphuric acid, and this increases the rate of decomposition of the mixture, the reaction thus becoming autocatalytic.

The basis of almost all pyrotechnic mixtures other than coloured lights is "meal powder," a fine grain gunpowder to which sulphur, nitre or charcoal, or two or all of these are frequently added to reduce its rate of burning.

Fireworks are almost invariably put up in paper cases, these being made by hand by pasting several layers of paper together. Rockets, squibs, crackers, etc., are all made by this means, and even the "shells" which are fired from mortars and contain coloured stars are made of paper, although they are sometimes as much as 2 ft. in diameter. These shells are made by tearing strips of paper and pasting them together inside a hemispherical mould, layer upon layer, until the desired thickness, ¼ in.–1 in., is attained. The hemisphere thus formed is then removed and dried, after which the edge is trimmed in a lathe and a complete shell formed by gluing two hemispheres together. The quality of the paper used is of great importance, and various qualities are used for different purposes. As a general rule, it should be free from all loading with mineral matter, and should have as great a mechanical strength as possible.

Coloured fire (Bengal fire) is the simplest form of firework, and simply consists of a moderately fiercely burning mixture containing suitable salts to impart colour to the flame, strontium and calcium being used to produce

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red, barium or copper for green, etc. Many different mixtures can be used, of which the following may be considered typical:—

Red.		Yellow.		Green.		Blue.	
$KClO_3$	78	$NaNO_3$	70	$Ba(NO_3)_2$	66	$KClO_3$	45
$SrCO_3$	15	S	20	Sugar	33	C	5
Shellac	7	Sb_2S_3	7	Shellac	1	$CuCO_3$	10
		Carbon	3			HgCl	35
						Shellac	5

Stars are very similar in nature, but are contained in a rocket or shell, and only liberated and ignited when the rocket or shell has reached its maximum height. They are of two types, viz. "naked" or "pumped" and "pill-box." The former are composed of a mixture of carbon, sulphur, meal powder and a nitrate to impart the desired colour, the ingredients being mixed together with shellac, and then either moulded into pellets or spread out and cut up into cubes, after which the solvent is dried off and the "stars" loaded direct into the rocket or shell. "Naked" stars should only be employed in fireworks of the smallest sizes, as they are very apt to crumble. In any case, to avoid crumbling it is very important to use shellac or other binding material which is completely soluble in the solvent used, usually methylated spirit. As a rule no special device is used for igniting naked stars, ignition being brought about by the burster charge, and for this reason they are almost invariably composed of nitrate, as such mixtures are more readily ignited than chlorate mixtures, and in any case a naked chlorate star in contact with meal powder cannot be used, as it would mean having a chlorate in contact with sulphur. Sometimes, however, the moulded pellets are made with a perforation into which a piece of quick match is inserted to assist their ignition.

"Pill-box" stars are much safer than "naked" stars, and are put up in paper cases, ignition being brought about by means of quick match. They are usually chlorate mixtures, *e.g.*—

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	Red.			Blue.	
$KClO_3$	47	56	$KClO_3$	40	37
Sugar	21	23	Sugar	25	25
$SrCO_3$	22	10	Cu_2S	15	—
HgCl	—	11	Cu	—	6
			HgCl	20	32

Green.		Yellow.	
$KClO_3$	26	$KClO_3$	59
Sugar	22	Na Oxalate	17
$Ba(NO_3)_2$	30	Shellac	24
HgCl	22		

Magnesium or aluminium powder is also sometimes added in order to increase the brilliancy.

Floating Stars are very similar, but are attached to a parachute of silk or soft paper. They are made to burn slowly, and frequently the pellets are made up of several layers, each layer of a different composition, so that they change colour as they burn.

Star Shell for military purposes is composed of barium and potassium nitrate, magnesium powder, paraffin and boiled linseed oil. The composition used by the British Army is made up of—

$Ba(NO_3)_2$	1 lb. 11 oz.
KNO_3	1 lb. 2 oz.
Mg	1 lb. 3 oz.
Paraffin	5 oz.
Boiled oil	3 per cent.

Whereas the magnesium light rocket is made up of—

$Ba(NO_3)_2$	1 lb. 8 oz.
$KClO_3$	1 lb. 2 oz.
Mg	1 lb. 8 oz.
Boiled oil	3 per cent.

the propellant charge being meal powder.

Squibs are paper cases with a small exploding charge at one end, and above it a long layer of slow burning composition composed of meal powder mixed with sulphur, or sulphur

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and charcoal. They are primed with a little gunpowder, and when ignited burn fiercely and then explode when the ignition spreads to the charge of gunpowder at the end.

Catherine Wheels are long, narrow paper tubes filled with a mixture of meal powder (2 parts) and potassium nitrate (1 part) and sulphur (1 part), and then wrapped round a circular wooden disc. When a pin is placed through the centre of the disc to act as a spindle and one end of the paper tube ignited, the escaping gas causes the wheel to revolve.

Roman Candles are paper cases containing coloured stars separated by layers of a rather fiercely burning composition of meal powder, nitre sulphur and charcoal, such as—

Meal powder	3	4	8
KNO_3	4	5	2
S	1	4	2
C	2	1	3

This composition burns fiercely with a good display of sparks, the stars being ejected by the gases. Naked stars are generally used, and the composition must not be tightly packed, as otherwise they may break or crumble.

Whistling Fireworks are paper tubes packed with a mixture of three parts of potassium picrate and two parts of potassium nitrate. One end of the tube is left open and the other is closed, the gas escaping from the open end causing a loud whistling noise. They are usually used as a garniture for rockets or shells.

Rockets consist of two parts, viz. the body containing the propellant, and the head containing the garniture. The body is a paper tube constricted at the lower end either by squeezing and then tying with a ligature, or by means of a clay plug. In order to fill the body a conical mould is inserted through the constricted end, point upwards, and the propellant charge then added little by little and well malleted home. There is considerable skill required in

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doing this, as the packing must be quite even if good results are to be obtained. When the propellant has been added the top is closed with a perforated clay plug through which a piece of quick match passes in order to fire the head, and the conical mould then withdrawn. The head is another paper case containing a burster charge of gunpowder and garniture in the form of stars, floating stars or whistling fireworks, and is glued on to the body. The stick is then attached so that the rocket balances when supported about one inch from the base. It is fired by applying fire to the conical hole left in the base by the mould. The outrush of gases causes the rocket to ascend, and when at its maximum height the quick match causes the burster charge to explode, thus liberating the garniture. Various compositions are used for the propellant, such as the following :—

Meal powder	2	1	3
KNO_3	4	20	16
C	2	12	8
S	1	2	4

although iron filings are frequently added in order to produce a good train of sparks.

Life-saving Rockets are similar, except that they have, of course, no garniture, and are used for carrying a rope from shore to ships in distress. The propellant composition is usually made up as follows :—

KNO_3	7 lbs.
C	3¼ lbs.
S	2 lbs.

Sometimes a head is provided closely resembling the body, so that when the original body is exhausted the rocket obtains a fresh impulse due to the firing of the head.

Touch Paper is much used for igniting fireworks, and is made by brushing paper, usually blue in colour, on one side with a solution of potassium nitrate (half a pound to the gallon) and then drying. *Slow match* for pyrotechnic purposes is made by soaking blotting paper in lead nitrate solution (2½ lbs. per gallon) and, after drying, pasting the

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sheets together, usually so as to give six thicknesses. Pyrotechnic *quick match*, on the other hand, is made by impregnating lamp wick cotton with a smooth cream of hot starch solution and meal powder, and then dusting it over with dry powder. Where ordinary quick match is not sufficiently rapid it is threaded through paper tubes, as the confinement greatly increases its rate of combustion. Such tubes are known as *leaders*, and are used when various parts of a firework have to be ignited almost simultaneously.

Fog Signals for the use of lighthouses during fog are usually composed of Tonite, *i.e.* guncotton pulp that has been impregnated with potassium or barium nitrate, and then compressed into pellets and dried. They are fired by means of a detonator.

Photographic Flash Lights are required to give out light rich in actinic rays, and for this reason magnesium powder enters into the composition of most of them. The original flash lights were very simple in construction, and consisted of a little magnesium powder placed in the bowl of a clay pipe, a piece of cotton wool soaked in spirit resting lightly on the top of the bowl. The spirit was lighted and the magnesium powder then blown into the flame by blowing air through the stem of the pipe. At present it is usual to use magnesium powder mixed with an oxidizing agent such as potassium chlorate or perchlorate or barium chlorate, this latter salt giving a safer mixture than potassium chlorate. Attempts have also been made to utilize the fact that the oxides of the rare earth metals give off a great deal of light when incandescent (*cf.* incandescent gas mantles) and flash powders composed of magnesium powder mixed with thorium or zirconium nitrate have been placed on the market. Barium peroxide has also been used as an oxidizing agent, and collodion cotton is also employed to some extent as an ingredient.

In modern flash lights it is customary to replace part of the magnesium by the less expensive aluminium powder, and good results have been claimed with the following mixture :—

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Al	50
Mg	100
Fe_2O_3	30
$CuCO_3$	30
$MgSO_4$	5

As will be seen, the oxygen is supplied by the ferric oxide, so that this powder is closely akin to Thermit in nature. The anhydrous magnesium sulphate is added as a deterrent to prevent combustion becoming too rapid.

The *Holme's Life-buoy* is an ordinary life-buoy to which is attached a can containing calcium phosphide, this can being closed by a lightly soldered plug. When used this plug is wrenched out and the buoy then thrown into the sea. The water on coming into contact with the calcium phosphide causes an evolution of the spontaneous inflammable phosphides of hydrogen, the flame of which is visible at night. The charge is usually sufficient to burn about half an hour.

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PYROTECHNY

There seems to be no modern works dealing with Pyrotechny, but A. Bujard, "Leitfaden der Pyrotechnik," Stuttgart, 1889, gives a fair idea of the subject. An interesting article will also be found in Thorpe's "Dictionary of Applied Chemistry," but it requires to be read with care, as a large number of the compositions described are out of date and illegal.

See also the article on "Feuerwerkerei" in Ullmann's "Enzyklopädie der technischen Chemie," vol. v.

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Section VIII.—EXPLOSIVE PROPERTIES

Apart from analytical operations certain tests are applied to explosives in order to determine their properties. These tests may be roughly divided into two classes, viz. tests to determine the nature of the explosive and tests to determine its stability. The former includes tests for power, violence and velocity of detonation, pressure, heat and temperature of explosion, and production of flame, whereas the latter includes tests for chemical stability, of which the Abel heat test is the most important, and tests for sensitiveness to heat and mechanical shock. In addition, propellants are tested to determine the velocity of the projectile and the pressure attained in the gun, and, of course, these tests must be carried out with the type of arm for which the explosive is intended. Explosives for use in coal mines are further tested to determine their safety when fired in the presence of fire damp and coal dust, and these tests, together with the methods used for studying flame production, have already been described in Section V.

Tests for Power

The power of an explosive is its capacity for doing useful work, and must not be confused with violence or brisance, this latter being rather the rate at which work is done, or the shattering effect produced. It is impossible to measure the power of an explosive in definite units such as foot-pounds, but tests have been devised which allow the relative power to be compared. Comparisons should, however, only be made between explosives of a similar nature, as the figures given for brisant and non-brisant

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explosives are hardly comparable. Useful as these comparisons are, it should always be borne in mind that the best test of an explosive is its behaviour under working conditions, and it is practically impossible to imitate these. There are three methods in general use for testing power, viz. the Trauzl lead block test, the mortar test, and the ballistic pendulum. Of these the Trauzl lead block is the simplest, and is the most generally used on the Continent, whereas the ballistic pendulum is the most elaborate and probably the best. A ballistic pendulum, however, is an expensive piece of apparatus, so that its use is practically limited to the official testing of explosives for the British Permitted List or the United States List of Permissible Explosives.

Trauzl Lead Block.—This test is based on the volume of the cavity formed when a given weight of an explosive is fired in a bore-hole made in a block of pure lead, and in order to obtain comparable results the test must be carried out under absolutely standard conditions. These conditions have been defined by a committee appointed by the International Congress of Applied Chemistry (V. Congress, 1903, vol. ii. p. 256), and may be summarized as follows:—

The blocks are circular in section, and are 20 cm. high and 20 cm. in diameter. They are cast of pure, soft chemical lead, the bore-hole, which is 125 mm. deep and 25 mm. in diameter being situated in the centre and cast in by means of a suitable mould. Before use the blocks are allowed to stand for several days. The weight of the explosive used is 10 grams, and this is made up into a cartridge 25 mm. in diameter by wrapping it in a piece of tinfoil 70 mm. wide, the length of the long sides being 120 and 150 mm. The foil is of such a thickness that 1 square metre weighs 80–100 grams. A No. 8 electric detonator is inserted into the cartridge, and the cartridge then pressed to the bottom of the hole with a wooden rod, the wires being kept central. Tamping is then applied by filling the hole with sharp, dry quartz sand, which is of such a degree of fineness that it passes a sieve with 144 meshes per $cm.^2$, the wires being

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·35 mm. thick. The charge is then fired electrically, and any residue left shaken and brushed out of the cavity. The volume of water required to fill the cavity is then determined, and the volume of the original bore-hole deducted, the figure thus obtained being taken as a measure of the power of the explosive. At least three tests should be made, and the mean of the figures thus obtained compared with the mean of three similar tests made with a standard explosive of approximately the same velocity of detonation. The test should be carried out at a temperature of 15°–20° C. The enlargement caused by a detonator alone is sometimes determined in the same way,

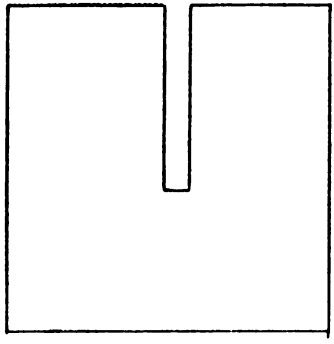

FIG. 26.—Trauzl Block before Firing.

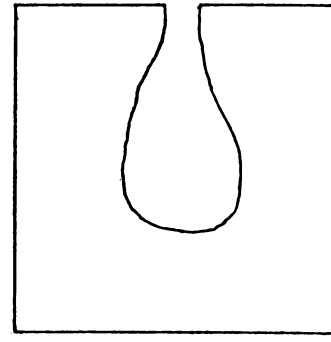

FIG. 27.—Trauzl Block after Firing.

and this value deducted, but as the test is only a comparative one this is unnecessary provided the standard explosive is fired with a detonator of the same strength. Figs. 26 and 27 show Trauzl blocks in section before and after firing.

The Trauzl block test is a convenient one, but suffers from several uncertainties. In the first place, it fails with non-detonating explosives, as the pressure is developed so slowly that the tamping is blown out. In the second place, it is affected by variations in the lead and in the sand used for stemming, so that as large a number of blocks as possible should be cast from one melt and a good stock of sand kept. With aluminium explosives, and other explosives with a

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very high temperature of explosion, high results are obtained owing to erosion, whereas with explosives containing an excess of oxygen the influence of the tinfoil wrapper comes into play unless the standard explosive used also contains a similar excess of oxygen. The weight of the wrapper is about 1 gram, *i.e.* 10 per cent. of the weight of the explosive. The following table gives a few characteristic results obtained when explosives are fired in the lead block :—

N.G.	540 cc.	P.A. ..	297 cc.
B.G.	530 cc.	T.N.T...	254 cc.
Dynamite No. 1 ..	300 cc.	Tetryl ..	375 cc.
Gelignite (65 N.G., 25 $NaNO_3$, 8 W.M.)	420 cc.	G.C. ..	290 cc.

Mortar.—This is shown in Fig. 28, the test being carried out by measuring the distance that the shot is thrown when

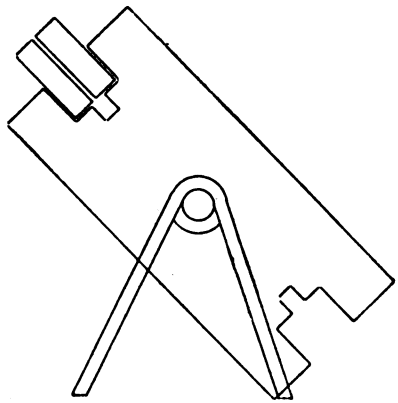

FIG. 28.—Mortar.

10 grams of the explosive are fired. The explosive is weighed out in a wooden shell 30 mm. in external diameter and 60 mm. deep, which is then inserted into the charge chamber. A piece of safety fuze with a detonator at one end is passed through the central hole in the shot, and the shot then placed in position with the detonator pushed well down into the explosive. The shot weighs 15 kilos. and should be a good

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but not a tight fit in the gun. The gun is securely fixed on a concrete foundation, and is usually set with its axis at an elevation of 45°. The distance that the projectile is thrown is measured and compared with that obtained with an equal weight of a standard explosive of the same nature. The bore at the back of the gun is provided so that when one end becomes worn out the gun can be turned.

The method is a somewhat crude one, but gives more constant results than one would expect, and is useful when experimenting with new explosive mixtures. In order to obtain reliable results at least three shots should be fired, and it is advisable also to fire one or two clearing shots first in order to free the gun from rust. When new the projectiles are inclined to expand in diameter owing to the shock of the explosions, and give unreliable results, but this expansion ceases after a few shots have been fired. In order to make a gas-tight joint the shot should be well lubricated before being inserted. The following figures give an idea of the average throw obtained with a few standard explosives, a 15 kg. shot being used, and the gun being set at an angle of 45°:—

Blasting Gelatine..	240 metres
Gelatine Dynamite	188 ,,
Gelignite	168 ,,
Dynamite No. 1	132 ,,

The explosives on the British Permitted List give throws usually varying between 80 and 120 metres.

In order to save the trouble of fetching the projectile after each shot, the gun is sometimes mounted as a pendulum and the shot fired into a sand bank a few feet away. In this case the recoil is measured, the arrangement being very similar to that of the ballistic pendulum described below.

The Ballistic Pendulum.—This is the Official Test for the power of coal mine explosives both in the United States and in this country, and both pendulums will be described. The test has the great advantage that a reasonable weight of explosive is used, ½ lb. in America and ¼ lb. in this country,

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and that it is fired in its original wrapper and hence all wrapper influence is eliminated, or, rather, the wrapper influence is much the same as it would be under working conditions.

The pendulum at the U.S.A. testing station at Pittsburg consists of a 12·2 in. U.S.A. Army mortar, weighing 31,600 lbs. It is slung in a stirrup made of 1½-in. machine steel rod, the rods being 89¾ in. long. It is suspended on nickel steel knife edges working on nickel steel bed plates, the bed plates resting on massive concrete pillars. The explosive is loaded into a steel gun which runs on a 30-in. gauge track and is tamped with one pound of clay, or two pounds when slow burning non-detonating explosives such as gunpowder are being tested. Before firing the gun is run up to the mortar until exactly $\frac{1}{16}$ in. away from it.

The charge of standard explosive is eight ounces of 40 per cent. American Straight Dynamite of the composition—

N.G.	40
$NaNO_3$	44
W.M.	15
$CaCO_3$	1

which gives a swing of 2·7–3·1 in. By means of trial and failure the weight of the explosive that gives approximately the same swing as this is then determined, and three shots fired with this weight. The exact equivalent is then calculated from the ratio—

$$S_x : S_D = W : \tfrac{1}{2}$$

where S_x = swing given by W lb. of the explosive under test,
S_D = swing given by ½ lb. of the standard explosive.

The pendulum in use at the British testing station at Rotherham is somewhat smaller. The pendulum itself is a mortar weighing 5 tons and suspended by steel rods from an overhead axle having roller bearings. The bore-hole of the gun is 30 in. long and 1⅞ in. diameter, and the gun is placed 2 in. from the mortar. Two pounds of well-rammed dry clay are used as stemming, and the charge of explosive

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used is four ounces. The swing given is read off and compared with the swing given by four ounces of Gelignite containing 60 per cent. of nitroglycerine, this swing being 3·27 in. The swings recorded by the most important explosives on the Permitted List are given in Section V.

Tests for Violence

By the violence or brisance of an explosive is meant the shattering effect produced. This is rather an illusive quantity and depends not only on the power but also on the maximum pressure developed, this in turn depending on the velocity of detonation. Proposals have been made to calculate the brisance mathematically, Bichel giving the formula $\frac{1}{2}MV^2$ where M is the mass of the gaseous products and V the velocity of detonation. These, and other similar expressions, however, do not give a satisfactory value, and attempts have been made to determine the comparative brisance experimentally. The results obtained cannot be said to be very satisfactory so far, but they do give some idea of the relative brisance. They are all based on the crusher gauge principle and measure the deformation of a cylinder of a soft metal, such as copper or lead. The best-known form of apparatus is the brisance meter devised by Kast and shown in section in Fig. 29. This consists of a hollow steel cylinder resting on a heavy steel base. A small copper cylinder is placed centrally on the base, and on the top of this a heavy steel piston which is an accurate fit in the steel cylinder. On the top of the piston is placed a nickel steel plate 20 mm. thick and weighing 320 grams, on which rest two 4 mm. lead discs. These lead discs are renewed after each shot, and have the object of protecting the steel plate. The explosive is fired on the top of them,

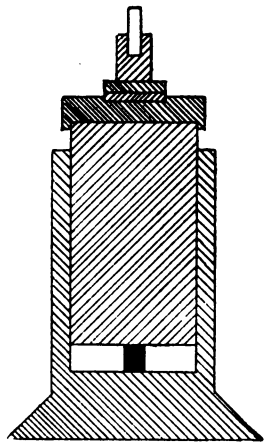

Fig. 29.—Brisance Meter.

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and the crushing of the copper cylinder taken as a measure of its brisance. The figures obtained vary with the diameter and length of the cartridges, and seem to be at a maximum when the length of the cartridge is about four times its diameter. As would be expected, they also vary with the density of the explosive, this having a great effect on the velocity of detonation. As the explosive is fired unconfined, the test does not in any way represent the conditions attained in practical blasting operations. The following figures were obtained using 15 grams of the explosive in the form of a cartridge 21 mm. in diameter :—

Explosive.	Density.	Velocity of detonation.	Brisance.
Tetryl	1·42	—	3·57
	1·53	7145	3·91
	1·59	7160	3·94
Picric Acid	1·34	6160	2·81
	1·40	6700	3·34
	1·53	7000	3·55
	1·60	7100	3·88
T.N.T.	1·34	5940	2·80
	1·45	6400	2·93
	1·50	6590	3·13
	1·60	6680	3·13
D.N.B.	·93	—	1·61
T.N.T. 30	2·65	4600	2·52
$Pb(NO_3)_2$ 70	2·75	4700	2·86

Velocity of Detonation

The velocity of detonation represents the speed at which detonation spreads or travels throughout the mass of the explosive when once initiated. It can be determined by two methods, viz. by the absolute method, in which the interval of time is actually measured, and by the comparative method, in which the velocity of detonation of one explosive is measured relatively to the velocity of detonation of another.

Owing to the high velocities attained, frequently 6000–8000 metres per second, the absolute method resolves itself into the problem of measuring very small intervals of time.

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For example, if a train of cartridges of Blasting Gelatine 10 metres long is detonated at one end, the detonation has reached the other end in about ·0013 second. For accurately measuring such very short intervals of time the best method has been found to be a drum of known diameter rotating at a known speed. From the speed of rotation and diameter of the drum the linear velocity of any point on the surface is readily calculated, and hence the time that

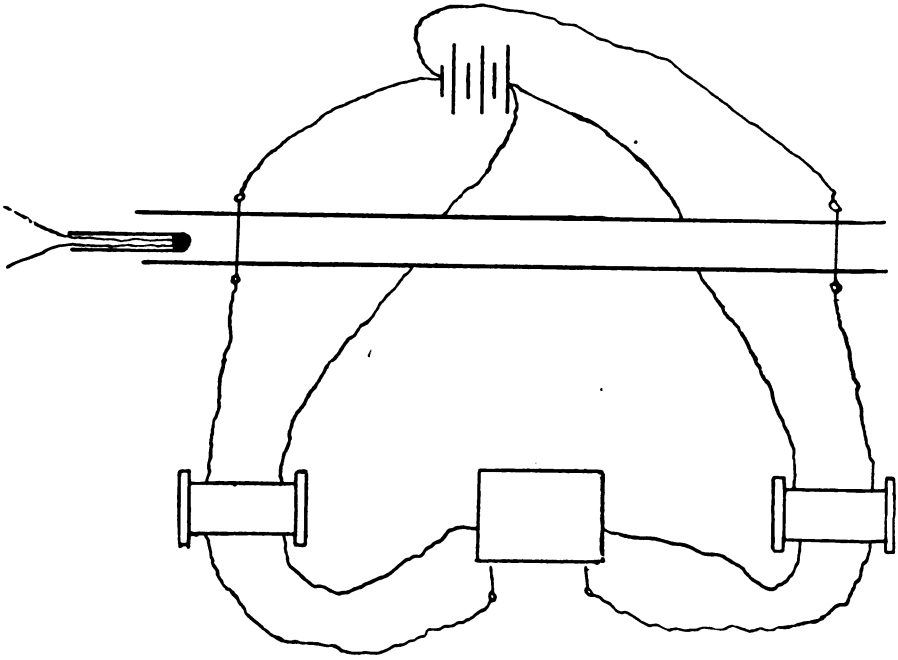

FIG. 30.—Determination of Velocity of Detonation (Mettegang's Direct Method).

has elapsed between the making of two marks can be deduced. The Mettegang time recorder for the determination of velocity of detonation is based on this principle and by its use intervals of time down to 1×10^{-7} seconds can be measured. The arrangement of the apparatus is shown diagrammatically in Fig. 30. Two thin wires are embedded in the explosive under test at a suitable distance apart, usually about 3–4 metres, each of these wires forming part

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of the primary circuit of different induction coils. One terminal of the secondary circuit of each of these coils is connected with the axis of the drum, while the other terminal is connected with a platinum point which nearly, but not quite, touches the smoked surface of the drum. The drum is rotated by an electric motor at such a speed that the linear velocity of the surface is 100 metres per second, and the explosive is then detonated. When the detonation reaches the first wire it breaks the primary circuit in one of the coils, and the current thereby induced in the secondary circuit causes a spark to pass between the corresponding platinum point and the drum, this spark leaving a mark on the smoked surface. The same thing takes place when the detonation reaches the second wire, and by measuring the distance between the two marks the time taken for the detonation to travel between the two wires is calculated. The distance between the two marks on the drum is measured by a travelling microscope with a vernier scale reading to ·01 mm., this corresponding to 1×10^{-7} seconds when the surface velocity of the drum is 100 metres per second. By using more wires, each wire being connected with a separate coil and platinum point, measurements of the velocity of detonation at different points in a long train of cartridges can be determined, thus ascertaining in any given case whether the velocity remains constant or whether it increases or decreases.

A certain amount of time always elapses between the breaking of the primary circuit and the passing of the spark due to the induced current in the secondary circuit. In order to eliminate error from this source by making the lag the same in both cases, the strength of the current in both primary circuits must be the same, the coils must be of exactly similar construction and resistance, and the direction of the flow of the current must be the same in both.

In the indirect method the velocity of detonation of the explosive is measured in terms of the velocity of detonation of another explosive, detonating fuze always being chosen for purposes of comparison owing to its convenience and very

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constant velocity, about 5000 metres per second. The determination is a very simple one, the arrangement used being shown in Fig. 31. Two detonators are inserted into the cartridge under test at a measured distance apart, 10 cm. being sufficient. Each detonator is connected to one end of a length of detonating fuze, the centre of which rests on a lead plate. A mark is made on the lead plate corresponding with the middle of the length of fuze, and the cartridge of explosive then fired by means of an electric detonator inserted in one end. When the detonation reaches the first detonator it detonates one end of the fuze, and when it reaches the other detonator it detonates the other end of

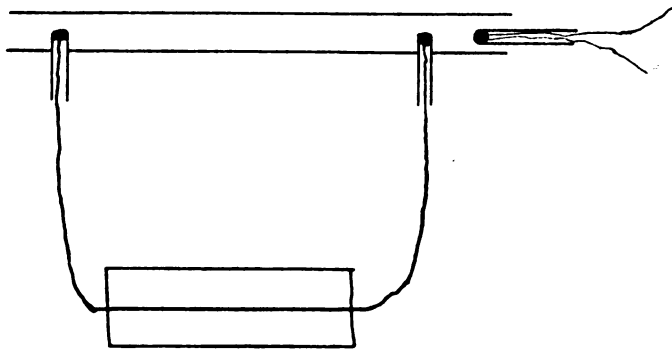

FIG. 31.—Determination of Velocity of Detonation (Indirect Method).

the fuze. These two waves of detonation travelling through the fuze in opposite directions meet at a certain point, and where this happens a mark is left on the lead plate. Obviously the distance of this mark from the centre of the fuze represents the start that the one wave of detonation had over the other wave, and hence the velocity of detonation of the explosive can be calculated in terms of the velocity of detonation of the fuze.

The velocity of detonation is affected to some extent by the diameter of the cartridge, and increases as the diameter increases. It usually, however, reaches a limiting value above which it remains constant however much the diameter is increased. With pure nitroaromatic compounds maximum

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velocity is reached at 10 mm., but with other explosives it is somewhat more, usually about 30 mm., and in the case of ammonium nitrate explosives is frequently over 50 mm. The following figures serve to illustrate this point :—

Explosive.	Diameter (mm.).	Velocity (metres per sec.).
Pure cast T.N.T.	21	6700
D = 1·58	75	6595
,, ...	220	6675
,, ...	300	6710
Aldorfit $AmNO_3$ = 81	—	—
D = 1·15 T.N.T. = 17	26	4430
Rye meal 2	40	4925

The strength of the detonator or primer has little or no effect provided it is sufficiently strong, but very irregular results are sometimes obtained at first, both abnormally high and low. With most explosives the velocity of the wave of detonation increases or decreases until its normal value is reached and then remains constant, but insensitive ammonium nitrate explosives behave in a somewhat different way, the velocity gradually decreasing. This is probably due to insensitiveness, as the effect is most marked when the explosive is compressed to a high density, in which case detonation is usually difficult to start. Lowering the temperature, on the other hand, has no effect, velocities remaining constant even at the temperature of liquid air (—180° C.).

The effect of compressing the explosive and thus raising its density is very great, and experiments seem to demonstrate that velocity of detonation is a linear function of density. It is true that with Cheddite and some other explosives the velocity falls if the density exceeds a certain figure, but this is probably due to the difficulty in starting a satisfactory wave of detonation owing to the diminished sensitiveness of the explosive. Thus picric acid enclosed in paper cartridges 20 mm. in diameter gave the following figures when fired with a detonator consisting of ·5 gram

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fulminate and a primer containing 25 grams of Dynamite No. 1—

Density.	Velocity.
·93	5035
1·31	6255
1·46	6988
1·67	7277
1·71	5045
1·74	Failed

By increasing the primer charge to 80 grams of Dynamite picric acid gave the following figures:—

Density.	Velocity.
1·62	7370
1·72	7490
1·73	7645
1·74	7645

showing that by increasing the initial shock the explosive could still be detonated and that the velocity of detonation continued to increase.

Very similar results were obtained with T.N.T.—

Density.	Detonator.	Primer.	Velocity.
·84	·5 gram fulminate	Nil	3822
·91	,,	,,	4087
·92	,,	,,	4170
·90	,,	25 grms. Dynamite No. 1	4170
·91	,,	,,	4170
1·32	,,	,,	6217
1·46	,,	,,	6675
1·56	,,	,,	6880
1·59	,,	,,	7056
1·60	,,	,,	7140
1·605	,,	,,	Failed
1·61	,,	80 grms. Dynamite No. 1	6943
1·71	,,	,,	7800

The last figure was obtained when the T.N.T. was confined in a copper tube, whereas the others were obtained when the explosive was confined in paper.

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The influence of the degree of confinement is very slight with sensitive explosives having a high velocity of detonation, but with the insensitive explosives of the ammonium nitrate class it is very considerable. Figures illustrating this will be found on pages 194 and 195.

Nitroglycerine exhibits the peculiarity of having two velocities of detonation, one about 1300–1500 metres per second, and the other about 7000–9000 metres per second. At which of these velocities the wave of detonation travels depends a good deal on the diameter of the tube and on the strength of the primer or detonator, the following results having been recorded:—

Detonator.	Primer.	Diameter.	Velocity.
1·6 grams.	—	6 mm. (glass)	Failed
1·6 ,,	—	9 mm. (glass)	654
1·6 ,,	—	25 mm. (iron)	1441 and 7690
1·6 ,,	—	38 mm. (iron)	8527
·8 ,,	—		1776
—	Detonating fuze	38 mm. (iron)	7234

Interesting results of the same nature have been obtained with various American Dynamites and gelatinized explosives. These latter when fired with a detonator only have a velocity of detonation of about 2300 metres per second and this figure is scarcely altered by increasing or decreasing the amount of nitroglycerine present. When fired with a primer of American Straight Dynamite, however, the velocity is much greater and increases with the percentage of nitroglycerine, being about 5100 metres per second with a nitroglycerine content of 40 per cent., 6600 metres with Gelignite containing 60 per cent. of nitroglycerine, and 7000 metres with 75 per cent. Gelatine Dynamite. With the Straight Dynamites, on the other hand, the velocity of detonation increases regularly with increasing nitroglycerine content, and is not affected by the use of a primer, provided a detonator of suitable strength is used.

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AMERICAN DYNAMITES

Per cent. N.G.	Straight Dynamites with or without Primer.	Gelatines.	
		Detonator only.	Detonator and Primer.
5	1294	—	—
10	2103	—	—
15	3095	—	—
20	3197	—	—
25	3296	—	—
30	4172	2484	—
35	4605	2278	—
40	4848	2230	5122
45	5032	2279	5544
50	5348	2355	5862
60	5973	2104	6606
75	6265	2165	6999

With Ammonia Dynamite in which half the nitroglycerine has been replaced by ammonium nitrate the velocity of detonation increases from 2100 with the 10 per cent. grade to about 4400 with the 45 per cent. grade and thereafter falls, the 60 per cent. grade giving a velocity of 3000. These explosives are not gelatinized, and the velocity is not materially increased by the use of a primer. Obviously the increase in velocity shown when passing from grade 10 to grade 45 is due to the increasing nitroglycerine content, whereas beyond 45 per cent. the ammonium nitrate content becomes the predominating influence, ammonium nitrate explosives always being slow.

The dual velocity of detonation exhibited by liquid nitroglycerine and gelatinized explosives is probably to be accounted for by their exhibiting dual elasticity, under a light shock behaving as liquids and under a heavy shock as solids. The velocity of sound depends on the modulus of elasticity of the transmitting medium according to the equation—

$$v = \sqrt{\frac{\mu}{d}}$$

where μ is Young's modulus and d the density, and probably the velocity of detonation follows a similar law,

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but no results seem to have been recorded in which the modulus has been determined. Certainly the velocity of detonation increases with the density instead of decreasing, but no conclusions can be drawn from this fact until the effect of increasing density on elasticity has been studied; for if the modulus increases more rapidly than the density, then the velocity of detonation would also increase if it followed the above equation. The following table gives the velocity of detonation in metres per second of a few well-known explosives, where irregular results have been obtained, the mean of the most concordant figures being given:—

Explosive.	Density.	Velocity.	Remarks.
Mercury Fulminate ..	—	3920	6·45 mm. diam.
Detonating fuze	—	5000–6000	Phlegmatized fulminate.
,, ,, ...	1·5	4800–5000	T.N.T. 4·4 mm. diam.
Guncotton, dry	·9	3900	—
,, ...	1·2	4300	—
,, ...	1·4	4800–6200	—
,, wet (15 per cent. H_2O)	—	5500–5800	23 mm. diam.
Nitromannitol	1·5	7000	4 ,, ,,
,, ...	1·9	7700	,, ,,
Nitroglycerine	—	1440 and 7690	25 ,, ,,
Tetryl	1·53–1·63	7200	21 ,, ,,
Picric acid	1·34	6160	,, ,,
,, ...	1·46	6700	,, ,,
,, ...	1·53	7000	,, ,,
,, ...	1·60	7100	,, ,,
Trinitrochlorbenzole ..	1·66	6800	,, ,,
Hexanitrodiphenylamine	1·58–1·67	7125	,, ,,
Trinitrocresol	1·52	6620	,, ,,
,, ...	1·62	6850	,, ,,
Trinitrobenzole (1.3.5)	1·63	6850	,, ,,
Trinitrotoluol (2.4.6) ..	1·34	5940	,, ,,
,, ..	1·45	6400	,, ,,
,, ..	1·50	6590	,, ,,
,, ..	1·60	6680	,, ,,
Dinitrobenzole (1–3) ..	1·50	6000	,, ,,
Macarite {T.N.T. 30 / $Pb(NO_3)_2$ 70 ..	2·73	4695	,, ,,
	2·89	4860	,, ,,
Tetryl 63 / D.N.B. 37 } compressed ..	1·58	6965	,, ,, 7155 *
T.N.T. 90 / D.N.T. 10 } compressed ..	1·56	6725	,, ,, 6680 *
T.N.T. 90 / D.N.T. 10 } cast	1·57	6565	,, ,, 6680 *

* These figures refer to the velocity of detonation of the chief constituent of the mixture at the same density.

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Explosive.	Density.	Velocity.	Remarks.
T.N.T. 50 D.N.T. 50 } compressed ..	1·53	6280	21 mm. diam. 6660*
T.N.T. 50 D.N.T. 50 } cast	1·51	1480 (Incomplete)	,, ,,
T.N.T. 50 D.N.T. 50 } cast	1·53	1910 (Incomplete)	,, ,,
T.N.T. 99·5 Soft wax ·5 } compressed	1·30	Failed	26 ,, ,,
	1·33	5515	,, ,, 5920*
Dynamite No. 1 { N.G. 75 Guhr 15	·63	1990	20 ,, ,,
	·85	2560	,, ,,
,,	1·34	3670	,, ,,
,,	1·54	5230	,, ,,
,,	1·62	6800	,, ,,
Blasting Gelatine { N.G. 92 C.C. 8	1·63	7700	30 ,, ,,
Gelignite { N.G. 62 .. C.C. 3 .. $NaNO_3$ 27 . W.M. 8 ..	1·59	2055	26 ,, ,,
	1·67	7000	30 ,, ,,
Dynamite, 50 per cent. { N.G. 50 .. Guhr 7 .. KNO_3 33·1 W.M. 9·7 $CaCO_3$ ·2	1·56	4610	26 , ,,
Dynamite, 40 per cent. { N.G. 40 .. $NaNO_3$ 42 W.M. 16·7 $CaCO_3$ 1·2	1·56	4440	,, ,,
Extra-Carbonite { N.G. 35 .. C.C. ·3 .. $Ba(NO_3)_2$ 4 KNO_3 25·5 Tan meal 34·8 Na_2CO_3 ·5..	1·13	3970	,, ,,
	1·20	4070	,, ,,
Carbonite No. II. { N.G. 30 .. $NaNO_3$ 24·5 .. Flour 40·5 .. $K_2Cr_2O_7$ 5 ..	1·05	3720	,, ,,
	1·10	3850	,, ,,
Wetter-Dynamite No. I { N.G. 30 .. $NaNO_3$ 31 .. Flour 30 .. W.M. 6 .. Naphthalene 2 Alum 1 ..	1·16	3900	,, ,,
Donarite { N.G. 3·8 .. C.C. ·2 .. $AmNO_3$ 80 .. T.N.T. 12 .. Flour 4 ..	·89	3700	26mm. diam. confined.
	1·31	4140	30 ,, ,,
	1·31	3930	,, unconfined.
Ammon-Carbonite { N.G. 4 .. $AmNO_3$ 82 KNO_3 10 .. Flour 4 ..	1·06	3385	40 mm. diam.
	1·23	3315	26 ,,
	1·19	1650	30 ,, unconfined.
	1·19	3100	30 ,, confined.

* These figures refer to the velocity of detonation of the chief constituent of the mixture at the same density.

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Explosive.		Density.	Velocity.	Remarks.
Aldorfit	$AmNO_3$ 81 ..	1·1	4825	40 mm. diam. confined.
	T.N.T. 17 ..	1·14	4410	26 ,, ,,
	Rye flour 2 ..	1·16	4925	40 ,, ,,
		1·17	4960	50 ,, ,,
Gesteins Dorfit	$AmNO_3$ 66 ..	1·10	3940	26 ,, ,,
	T.N.T. 15 ..	1·11	4420	40 ,, ,,
	KNO_3 5 ..	1·15	4045	26 ,, ,,
	NaCl 10 ..	1·15	4505	40 ,, ,,
	Rye flour 5 ..	1·17	4605	50 ,, ,,
Thunderite	$AmNO_3$ 92 ..	1·07	3650	30 ,, ,,
	T.N.T. 4 ..	1·07	2137	30 ,, unconfined.
	Flour 4 ..			
Permonite	$AmNO_3$ 42·5			
	T.N.T. 10 ..	1·05	3690	40 mm. diam.
	$KClO_4$ 32·5 ..	1·13	3780	,, ,,
	Starch 12 ..			
	W.M. 3 ..			
Ammonal	$AmNO_3$ 44·9			
	T.N.T. 31 ..	1·68	4850	21 ,, ,,
	Al 24·1 ..			

The velocity of detonation of Cheddite was given on page 110.

Pressure of Explosion

The exceedingly high pressures attained during explosion render exact measurement a matter of considerable difficulty. The first real instrument devised to estimate these pressures was the Rodman gauge, in which the pressure drove a hardened steel knife edge into a soft copper disc, the penetration being taken as a measure of pressure attained. The design of this gauge was afterwards improved by Nobel, who used a copper cylinder placed between a fixed and a movable steel piston. The pressure of the gases acting on the base of the movable piston crushed the copper cylinder, from the deformation of which the pressure could be deduced. This type of gauge is known from its mode of action as the crusher gauge, and is the standard method of measuring the maximum pressure developed by propellants in firearms. When used for this purpose the gauge is either screwed into a hole bored in the explosion chamber, or it is inserted behind the cartridge, so that the base of the cartridge is in contact with the movable piston.

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This latter point is important, as otherwise the momentum of the cartridge would cause abnormally high results. For the same reason the movable piston must be closely in contact with the copper cylinder, and when the gauge is screwed into the firearm the end of the piston on which the gases act, or the gas check which is usually placed in front of the piston, should be flush with the end of the cylinder in which the piston moves. Otherwise the momentum of the gases will cause abnormal crushing.

For use with rifled arms in which the maximum pressure attained is about 15 tons per square inch copper crushers are always used, but with shot guns in which much lower pressures, viz. about 3 tons per square inch, are developed, lead cylinders are frequently employed. The use of the crusher gauge is not, of course, confined to the study of propellants, but can be used for the study of blasting explosives when fired in bombs. It has the advantage of being inexpensive and convenient, but unfortunately its use is limited to the measurement of the maximum pressure attained, and it gives no information as to the rate of growth of this pressure.

Owing to the extremely rapid rate at which the pressure rises during explosion, records showing its growth are difficult to obtain, and although one or two instruments have been devised for the purpose their use is limited, and too much reliance must not be placed on the results obtained with them. One of the most satisfactory is the Bichel recorder. This consists of a massive steel cylinder, the internal dimensions of which are 20 cm. in diameter and 48 cm. deep, the capacity thus being 15 litres. Charges of 100–200 grams are fired, and the pressure attained recorded on a rapidly revolving drum by means of a piston acting against a powerful spring. The shape of the curve gives a qualitative idea of the rate of development of pressure, but the maximum is very much exaggerated owing to the momentum of the moving parts. By neglecting this peak value and extending the curve backwards by extrapolation until it cuts the ordinate corresponding

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to the moment of explosion a more nearly true value for the maximum pressure can be read.

Owing to the low density of loading the cooling influence of the walls is very great, and Bichel has sought to eliminate this by using two bombs, one of 20 litres capacity and one of 15 litres capacity. By means of inserting metal cylinders of different sizes into the larger of these he was able to obtain surfaces of 3600, 6600, and 7600 cm.^{2}, while retaining the same volume of 15 litres.

On firing charges under these three conditions he obtained maximum pressure values which when plotted out against the area were found to lie on a straight line. By extending this line to the point area = 0 the pressure with the surface eliminated could be read off. More recently Peteval has introduced an improved recording manometer in which a very stiff spring is used, and consequently only a very slight movement of the piston takes place, this being magnified by means of a mirror throwing a spot of light on to a rapidly moving strip of sensitized paper. This recorder has been used for the study of propellants, but not for high explosives.

Heat of Explosion

For determining the heat liberated on explosion apparatus similar to that used in ordinary calorimetric work is used, but with suitable modifications to enable it to withstand the high pressures attained. Two types of bombs are in general use, viz. the comparatively small bomb, the walls of which are very thick to enable it to withstand the high pressures developed, and the large bomb calorimeter in which the increased volume results in lower maximum pressures, and consequently less mechanical strength being called for. Berthelot and Sarrau both used small, light bombs which could only deal with a few grams of explosive owing to their lack of mechanical strength, and Nobel and Abel used bombs of about 32 and 119 c.c. capacity, these taking charges of 12 and 26 grams of black powder respectively. Nobel, however, also used a very heavy bomb in which as

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much as 500 grams of black powder could be exploded, although the capacity of the bomb was less than half a litre. It is very difficult, however, to construct bombs of this type suitable for dealing with large charges of modern high explosives, although a nickel steel bomb of 45 c.c. capacity taking a charge of about 20 grams has been used at the Neubabelsberg testing station, and is said to give satisfactory results.

In determining the heat of explosion it is advisable to use large quantities of the explosive in order to reduce the error due to the rather uncertain correction which has to be applied to eliminate the influence of the detonator. The uncertainty of this correction is due to the fact that the heat liberated by a detonator depends on whether it is fired in an atmosphere rich in oxygen or not. If oxygen is present not only is the carbon monoxide liberated by the fulminate burnt to carbon dioxide, but the copper of the detonator case is oxidized to cupric oxide, and heat is also liberated by the combustion of the electric wires, etc., the detonator always being fired electrically. Thus it has been found that a No. 3 detonator fired in the absence of oxygen liberates only ·116 Cal. whereas in an oxidizing atmosphere it liberates ·791 Cal., and the amounts are of course greater with larger sizes. In order to obtain a bomb which has sufficient mechanical strength to withstand the explosion pressure of large quantities of high explosive without undue thickness of wall, the capacity must be increased. This has been done by Bichel and Mettegang, who have constructed a bottle-shaped steel bomb with a capacity of 30 litres. Its walls are about 13 mm. thick, and it weighs some 70 kilos. The usual charge of high explosive used is 100 grams, but in the case of black powder more than double this amount can be used with safety. All the usual precautions of calorimetry are applied, and a thermometer reading to ·001° C. is employed for observing the rise in temperature, as on account of the large heat capacity of the metal the rise in temperature of the surrounding water is only about 1° C.

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The figures thus obtained give the heat evolved when the explosive is detonated and the gases cooled to the ordinary temperature, and therefore include the heat evolved by secondary reactions, such as the condensation of the greater part of the water vapour produced, and the heat evolved by the oxides of the alkali or alkali earth metals combining with part of the carbon dioxide produced to form bicarbonates. Hence, in order to obtain the heat that would be actually evolved under practical working conditions corrections must be applied. The following figures give the heat liberated by the explosion of a few well-known explosives. The figures refer to major calories per kilogram of explosive :—

Name.	Composition.	Heat liberated.
Dynamite No. 1 ..	N.G. 75, Guhr 25	1170
Blasting Gelatine ..	N.G. 92, C.C. 8	1422
Gelignite	N.G. 63·5, C.C. 1·5, $NaNO_3$ 27·0, W.M. 8·0	1321
Donarite	N.G. 3·8, C.C. ·2, T.N.T. 12·0, $AmNO_3$ 80·0, Flour 4·0	836
Thunderite	T.N.T. 4, $AmNO_3$ 92, Flour 4	777
Carbonite	N.G. 25, KNO_3 30·5, $Ba(NO_3)_2$ 4·0, W.M. 40·0, Na_2CO_3 ·5	576
Gunpowder	KNO_3 75, C. 13, S. 12	574
Nitroglycerine ..	—	1471
Ammonium nitrate ..	—	365
Mercury Fulminate ..	—	407

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Calorimetric experiments can also be combined with measurement of the volume of gas liberated. This can be ascertained by direct measurement, but the large volumes render this method troublesome. An alternative method is to weigh the calorimeter before and after allowing the gas to escape, and then calculating the volume from the weight found together with the density. More usually, however, the pressure is measured when the temperature has become constant, and the volume at N.T.P. then calculated by the laws of Boyle and Charles.

Temperature of Explosion

Very little is known of the temperatures attained by explosion, and no attempts seem to have been made to measure them directly, although the difficulties of doing so by means of an optical pyrometer should not be insuperable, this method having been, in fact, used for testing caps (see p. 152). It is theoretically possible to calculate the temperature attained from a knowledge of the total heat liberated and the specific heats and latent heats of vaporization of the products of explosion. Unfortunately two factors militate against the accuracy of such calculations. In the first place, nothing is known of the equilibrium conditions from a chemico-dynamic point of view of the products of combustion. It is fairly certain that at the high temperatures reached both carbon dioxide and water are to a large extent dissociated into their elements—

$$2CO_2 \rightleftarrows 2CO + O_2 \rightleftarrows 2C + 2O_2$$
$$2H_2O \rightleftarrows 2H_2 + O_2$$

and probably this dissociation to some extent goes a step further, molecular oxygen and hydrogen breaking down into the monatomic state. In all the above cases the high pressure attained tends to force the equilibrium to the left side of the equation, but nothing is known as to the composition of the equilibrium mixtures under the conditions

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attained during or immediately after explosion, and the same applies to other side reactions, such as the temporary production of methane. These side or consecutive reactions do not, of course, affect the total heat produced, but they do affect the temperature attained by causing the explosive reaction to take place over a greater time. In other words, side and consecutive reactions produce a lower temperature, but maintain this temperature for a greater time, the heat evolved being the same as if the reaction had been a straightforward one taking place in one step.

In the second place, the specific heats of gases are not even approximately constant quantities, but increase with increasing temperature, and it is impossible to determine them at the high temperatures attained during explosion, especially as these temperatures are unknown. Mallard and Le Chatelier have investigated the matter, and have deduced the following values (minor calories) for the molecular heats at constant volume of gases at temperature $t°$ C. :—

Permanent gases, such as N_2, O_2, H_2 and CO	$4{\cdot}80 + {\cdot}0006t$
Easily condensed gases, such as CO_2 and SO_2	$6{\cdot}26 + {\cdot}0037t$
Water vapour	$5{\cdot}61 + {\cdot}0033t$

and other investigators have obtained similar figures. To what extent these figures are accurate it is difficult to say, but it is improbable that their accuracy extends beyond 1500° C., whereas most temperatures of explosion are probably nearer 3000° C. The values of Mallard and Le Chatelier have, however, been adopted by the French Government for the calculation of the temperature of explosion of safety coal mine explosives. The calculation is a somewhat cumbersome one, but is quite straightforward, and as the temperatures of explosion of explosives for use in coal seams must not exceed 1500° C., or 1900° C. in the case of explosives for use in rock in coal mines, the results are probably more or less accurate, at all events, as far as the former are concerned.

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Chronography

In the case of propellants the velocity imparted to the projectile by the explosive under conditions of use is a most important point. The velocity of the projectile at various points in its trajectory is of the utmost importance in gunnery, but once the projectile has left the muzzle the influence of the propellant ceases, so that from the explosive manufacturer's point of view it is the mean muzzle velocity, usually abbreviated to M.M.V., that matters. The name ballistics is given to the study of the flight of projectiles, internal ballistics dealing with their behaviour when in the barrel, and external ballistics dealing with their flight from muzzle to target. The subject of ballistics is highly mathematical in nature, and no attempt will be made to treat it beyond very briefly indicating the general principles on which the experimental determination of velocity is based.

Instruments for determining the velocity of a projectile are known as chronographs, and, generally speaking, are of two types, viz. those in which the time is measured in terms of the time taken for something else to happen, *e.g.* in the time taken for a weight falling freely to fall a certain distance, and those in which the time is recorded directly. As the muzzle velocity attained by projectiles from modern high velocity rifled arms is usually from 2000 to 2500 ft. per second, the time recorder must be capable of recording small intervals of time. The passage of the projectile over the space in which its velocity is being determined is indicated by placing thin, brittle copper wires in its path, the projectile severing these wires and thus breaking an electric circuit so arranged that this interruption is recorded.

Le Boulengé's Chronograph is shown diagrammatically in Fig. 32. The fracture by the projectile of the first wire breaks the circuit of the electro-magnet A, and allows the weight B to fall. This weight is in the form of a tube sliding freely over a vertical rod, so that it can fall freely in an upright condition, but cannot topple over. To avoid

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all chance of friction between the tube and the rod the latter is sometimes omitted, but in this case it is difficult to be certain of the weight falling true. The fracture by the projectile of the second wire breaks the circuit of the magnet C, and allows the weight D to fall on to the button E. This latter is connected with a trigger and spring arrangement, not shown in the diagram, so that when struck it releases the knife point F, which, being actuated by the spring, strikes the falling weight B, and leaves a mark on its soft metal covering. By holding C with this mark in contact with F and then measuring the distance between the top of the weight and the bottom of the electro-magnet A, the distance which the weight has fallen can be found, and from this the time calculated. There are, however, several sources of error in the figures thus obtained, and these must be corrected. In the first place, there is the lag in the demagnetization (hysteresis) of the two magnets, which may or may not be the same; and in the second place, there is the time taken by the weight D in reaching E and the time occupied by F in springing forward. These errors are best allowed for by determining a zero value, this being done by breaking the two circuits simultaneously. When this is done the distance fallen by B represents the magnitude of the lag due to electrical and mechanical delays, and is to be allowed for when calculating velocity.

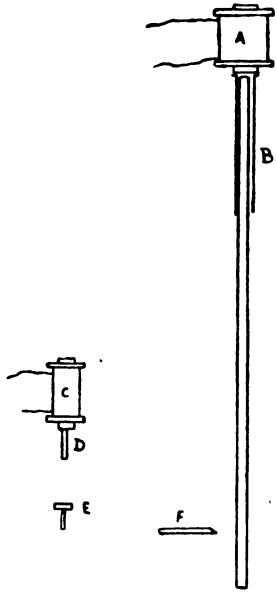

FIG. 32.—Le Boulengé Chronograph.

This instrument is simple to use, and gives very fair results over short distances, but when measuring a comparatively large interval of time the experimental error becomes big. This is due to the fact that when a body

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falls under constant acceleration, in this case the acceleration due to gravity, the space traversed varies as the square of the time—

$$S = \tfrac{1}{2}ft^2$$

or taking the value of f as 32 in foot-seconds units—

$$t = \frac{\sqrt{s}}{4}$$

So long as S is small, slight experimental error does not affect t to any great extent, but when S becomes comparatively large experimental error affects t severely.

To overcome this difficulty Le Boulangé devised an instrument which he named the Klepsydra, and which was based on the sand-glass principle. It consisted of a T-shaped vessel filled with mercury, the foot of the T being provided with a needle valve. This was actuated electrically, the breaking of the first wire opening the valve, and the breaking of the second wire closing it. By weighing the mercury which ran out in the interval the time during which the valve was open could be deduced. Zero value was obtained by breaking both circuits simultaneously.

The *Bashforth Chronograph* is based on a different principle, the time being measured by the beat of a pendulum. It consists of a revolving drum covered with smoked paper and geared to a board in such a way that as the drum revolves the board descends in a vertical direction. The board carries two styles which are held away from the surface of the smoked paper by means of electro-magnets. The circuits of these magnets are broken in succession by the projectile, thus allowing the styles to spring forward and mark the smoked paper on the drum. By means of a similar arrangement the pendulum makes time marks on the paper so that the time that has elapsed between the breaking of the two circuits is readily read off.

As in Le Boulangé's instrument, electrical and mechanical lag is corrected for by breaking the circuits simultaneously.

The *Schultze-Marcel-Dieprez Chronograph* is somewhat similar in construction, the breaking of the circuits of

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electro-magnets causing points to impinge on smoked paper attached to a revolving drum. In this instrument, however, time is marked simultaneously as a sinuous curve by means of a tuning fork of definite note, a style being attached to one prong and this style being in contact with the revolving drum. The tuning-fork is one of the most convenient methods of recording moderately short intervals of time, and is widely used in all branches of physics. As a matter of convenience the fork may be electrically controlled.

The *Mahieu Chronograph* is very similar to the above, but the drum in addition to a rotatory movement has also a longitudinal movement. The pens are in permanent contact with the surface of the paper but are attached to springs so that they spring to one side when the circuits of the electro-magnets are broken, thus causing a sudden momentary change in direction in the line being traced. The gearing controlling the relative longitudinal and rotatory movement of the drum is capable of alteration so as to render the instrument suitable for measuring high and low velocities and of dealing with both short and long flights.

Of course, either the Bashforth, the Schultze or the Mahieu instrument can be used for studying the velocity of a projectile at different points of its flight, it being only necessary to add more pens, but the Mahieu instrument is best adapted for work of this nature.

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Section IX.—SENSITIVENESS AND STABILITY

An explosive must fulfil certain conditions as regards sensitiveness and stability if it is to be suitable for commercial use. Unless intended for use as an initiator in percussion caps or detonators it should not be unduly sensitive to mechanical shock, as if it is it will be unsafe to handle and transport. At the same time it must be moderately sensitive to detonation and should be exploded fairly easily by influence, *i.e.* by the explosion of another cartridge of the same explosive lying near it but not actually in contact with it. A moderate degree of sensitiveness to explosion by influence is very desirable, as in blasting operations the bore-holes are usually charged with several cartridges which may be separated from one another by an air space due to defective ramming or by a layer of dust that has fallen into the hole during charging. Only one cartridge carries a detonator, and hence if the explosive is not readily exploded by influence some of the cartridges may fail to detonate and may cause an accident by being struck with a pick or shovel later on.

Stability is also highly desirable in an explosive intended for practical use, as should chemical decomposition take place during storage either the explosive may fail to detonate owing to loss of explosive properties, *e.g.* mercury fulminate, or it may become excessively sensitive or even explode spontaneously, *e.g.* chlorate-sulphur mixtures. All organic nitric esters have a tendency to decompose spontaneously at the ordinary temperature with the production of nitric acid. In most cases the reaction is an extremely slow one, but the velocity is greatly increased by the catalytic effect of acids.

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Since acid is actually produced by the decomposition the reaction is auto-catalytic, and hence it is of the utmost importance to test all explosives to determine the relative rate of production of acid. The Abel Heat Test is designed for this purpose, and in spite of its numerous imperfections gives a fair idea of stability. Chlorate-sulphur mixtures become dangerous from a similar cause, as atmospheric oxygen converts traces of the sulphur into sulphuric acid, this in turn liberating the highly dangerous chloric acid.

In addition to chemical stability, physical stability is also of importance. Thus most explosives containing nitroglycerine are liable to " sweat " or " exude " this substance, and as nitroglycerine is very sensitive to shock, the sensitiveness of the explosive is greatly increased although no chemical decomposition has taken place. Blasting gelatine is particularly liable to exude, exudation from Gelignite and Gelatine Dynamite being hindered by the absorptive properties of the wood meal used for doping these explosives. Deliquescence is another example of what may be termed physical stability. Explosives containing hygroscopic substances such as nitrate of ammonia take up moisture rapidly and may become quite unfit for use in a short time. The behaviour of an explosive when subjected to heat is also important, as is its behaviour when ignited, it being preferable from a safety point of view that under these circumstances it should burn away quietly without exploding. Finally, with powdered explosive mixtures degree of incorporation must be examined, as if not properly incorporated the constituents may separate when submitted to vibration, *e.g.* during transport, the explosive subsequently giving irregular results.

Mechanical Shock

Mechanical shock may be of two types, viz. shock due to friction or shock due to a direct blow. Comparative tests to determine the relative sensitiveness to friction of two explosives are difficult to carry out owing to the impossibility of fixing a standard of friction, but a rough idea can

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be obtained by various means such as grinding the samples in a mortar under as similar conditions as possible, striking them a glancing blow either by hand, or, better, by means of a pendulum arrangement.

A direct blow is much more easily imitated, and the falling weight test is invariably made use of. The apparatus for this consists of an anvil surrounded by a cylinder in which a piston works freely in a vertical direction, but is incapable of lateral movement. The explosive is spread on the anvil and the piston inserted into the cylinder so that its lower end rests on the explosive. The upper end of the piston is then struck a blow by a definite weight falling from a definite height, the height being gradually increased until explosion takes place. The falling weight slides freely over vertical guide rods so as to assure its falling true and striking a direct and not a glancing blow. The apparatus is shown in Fig. 33.

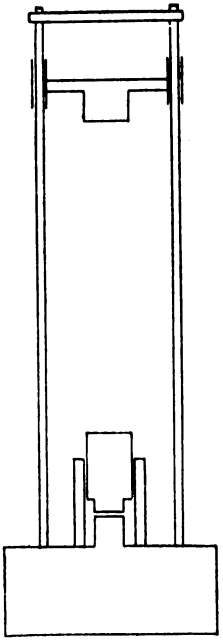

FIG. 33.—Falling Weight Test.

Theoretically the force of the blow is given by the mass of the falling weight multiplied by the velocity with which it strikes, or—

$$F = M \times \sqrt{2gS} \quad \text{or} \quad F = 8M\sqrt{S}$$

where M is the mass of the falling weight, S the height through which it has fallen, and g the acceleration due to gravity. In other words, the force of the blow should be proportional to the product of the weight into the square root of the height. In practice this is not the case, but the test is not of sufficient accuracy to be susceptible to mathematical treatment. For one thing, the results depend on the thickness of the layer of the explosive, its physical nature (density, size of grain, etc.), nature of the surfaces with which it is in contact, temperature, etc.; and hence,

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in order to obtain comparable results, the measurements must be made under strictly standard conditions. These have been laid down by an International Committee as follows :—

The anvil and the piston are made of hardened steel, the former resting on a firm foundation, and the area of each is 12·7 mm.^{2} (1·27 cm. in diameter). The weight of explosive used is ·05–·1, the amount being chosen so as to give a layer of standard thickness when spread uniformly on the anvil. The sample is dried over calcium chloride before use, and a fresh quantity taken for each experiment. The standard temperature is 18°–20° C., and the weight used weighs two kilograms.

Although these are the " Official " conditions they cannot as yet be said to be generally adopted, and most data available have been obtained under somewhat different conditions. The following results give a fair idea of the figures obtained, and were given by Will at the VI. Congress of Applied Chemistry. He used ·1 gram of explosive which had been dried at 40° C., and which was confined between steel surfaces and struck by a falling weight of 2 kilos. :—

Mercury Fulminate	2 cm.
Nitroglycerine (dry)	4 ,,
Lead picrate	5 ,,
Dinitroglycerine	7 ,,
Dynamite No. 1	7 ,,
,, (frozen)	25 ,,
Blasting Gelatine	12 ,,
,, ,, (frozen)	12–15 ,,
Gelatine Dynamite	17 ,,
Gunpowder (sporting)	70 ,,
,, (blasting)	85 ,,
Cheddite, Type 60	32 ,,
,, Type 41	36 ,,
Tetryl	40–65 ,,
Hexa-nitrodiphenylamine	40 ,,
T.N.T.	57–180 ,,
Dinitrobenzene	120 ,,
Tri-nitronaphthalene	175 ,,

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Ammonium picrate	80 cm.
Picric Acid	35–95 ,,
Guncotton (15 % H_2O)	85 ,,
Collodion (15 % H_2O)	100 ,,
Nitrocellulose propellants	30–54 ,,
Astralite	110 ,,
Donarite	110 ,,
Guncotton (20 % H_2O)	over 185 ,,
Collodion (20 % H_2O)	over 185 ,,

The following figures give a rough idea of the result of using different weights, in all cases ·1 gram of explosive being used, and this being wrapped in tinfoil. The falls are given in centimetres—

Explosive.	Composition.	·1 Kg.	·5 Kg.	1 Kg.	2 Kg.	5 Kg.
Guncotton ..	Dry	30–40	5–10	5–10	5–10	—
,, ..	15 % H_2O	—	15–20	10–15	10–15	5–10
Picric Acid ..	Crystalline	—	—	190–200	100–110	50–60
,, ,, ..	Compressed	—	—	—	140–150	80–90
T.N.T.	Crystalline	—	—	180–190	90–100	50–60
,, ...	Compressed	—	—	—	150–160	80–90
Carbonite No. 2	N.G. 30 $NaNO_3$ 24·5 Flour 40·5 $K_2Cr_2O_7$ 5·0	60–70	10–20	10–20	10–20	—
Donarite ..	N.G. 3·8 C.C. ·2 $AmNO_3$ 80·0 T.N.T. 12·0 Flour 4·0	—	—	120–130	60–70	15–20

The U.S. Bureau of Mines make use of a much larger machine in which greater quantities of explosive (20 grams) can be used. The falling weight weighs 200 kilos., and it is claimed that the figures obtained are of greater value, as actual practical conditions of use are more nearly imitated. The results obtained with this machine are quite different from those obtained by the smaller machines.

DETONATION

Sensitiveness to detonation depends largely on the physical state of the explosive undergoing test, and almost

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invariably decreases with increasing density, at high densities most explosives becoming so insensitive that they either cannot be detonated at all or, if detonated, the detonation is incomplete. At low temperatures sensitiveness is also diminished, while at elevated temperatures, as would be expected, the reverse is the case.

Ease of detonation is very simply determined by firing cartridges of the explosive with detonators of different power, and thus finding the weakest that will cause complete detonation under the conditions of the experiment. In the case of insensitive explosives that require a primer of some more sensitive explosive in order to detonate them completely the minimum weight of the primer is determined in the same way.

Influence

The capacity of one cartridge of an explosive to detonate another cartridge separated from it by an air space depends on the sensitiveness of the explosive and on the velocity of detonation, and hence also on the physical state of the explosive. It also depends on the nature of the surface on which the cartridges are resting, being at a minimum when they are suspended so that the wave of detonation can only be transmitted through the air, and at a maximum when the cartridges are resting on some hard, solid substance such as iron, in which case the wave of detonation is no doubt largely transmitted through the solid body. The distance through which detonation is transmitted is determined by the method of trial and failure, cartridges being exploded at increasing distances until the detonation is no longer transmitted from one to another. The figures thus obtained depend very much on the explosive, and vary from a small fraction of an inch to several feet. They also depend largely on the quantity of explosive involved, the explosion of large amounts, as for example during an accident, being capable of exploding other explosives at a considerable distance.

With reference to this experiments have been carried out to determine the velocity with which the wave of detonation is transmitted through air. This was done with

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detonating fuze in much the same way that velocity of detonation (page 187) is determined, two cartridges being separated from, but hanging end on to one another, and detonators inserted into the inner ends and connected by detonating fuze. One of the cartridges was then fired by an independent detonator, and the velocity of the wave of detonation through the air space determined by the behaviour of the fuze. It was found that the wave of detonation starts through the air with about the same velocity as it has in the cartridge, but that there is a retardation of about 50 metres per second for every centimetre of air traversed.

Heat and Ignition

The temperature at which an explosive ignites or explodes depends on how rapidly the temperature is raised, and is not a definite value like the melting-point of a pure chemical compound. It is rather analogous to the melting-point of a chemical compound in which decomposition sets in before its melting-point is reached, so that if the heating takes place slowly an indefinite value is obtained, whereas if the heating is rapid a fairly definite point can be determined at which the compound melts before decomposition has gone far enough to have much effect. Hence, in determining the ignition point of an explosive a preliminary experiment should first be carried out in which the heating is slow in order to determine an approximate value, and then a more accurate measurement made in which the apparatus is pre-heated to this point before the explosive is introduced. By this means it has been found that nitroglycerine explodes at about 170° C. and nitrocellulose at 10°–20° higher.

From the point of view of safety it is desirable that an explosive should burn away quietly without exploding if accidentally ignited, but the behaviour of an explosive under these circumstances depends largely on how the ignition is started, the degree of confinement of the explosive, and on the quantity. Hence laboratory results are of comparatively little value, although more or less

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useful indications can be obtained by igniting small quantities in various ways, such as by means of a match or a piece of safety fuze, or by throwing some of the explosive into a hot dish. As a rule, when in small quantity, nitroglycerine explosives burn away fiercely, but do not explode, but with larger quantities explosion generally takes place. Explosives containing chiefly ammonium nitrate and small quantities of nitroaromatic compounds, on the other hand, are almost incombustible, but large quantities submitted to a high temperature may explode.

Incorporation

Black powder and other explosives containing only solid constituents, such as ammonium nitrate explosives, if not sufficiently incorporated, may settle out when submitted to vibration during transport, and thereafter either do not explode at all or give very irregular results. To ascertain if the incorporation is satisfactory a sample of the explosive is submitted to vibration for a number of hours in a type of shaking machine. This is best constructed in such a way that the vessel containing the explosive is raised by a cam or sprocket wheel and lever and then allowed to drop back on to a metal stop. After a period of vibration the top layer and the bottom layer of the explosive are collected separately and analyzed.

Deliquescence

The capacity of an explosive to absorb moisture from the air is an important point, especially with those explosives which contain highly hygroscopic salts such as nitrate of ammonia or soda, as the absorption may reach such a point that the explosive can no longer be detonated, or in the case of nitroglycerine explosives the water taken up may displace the nitroglycerine and thus give rise to danger. The deliquescence of an explosive depends chiefly on its constituents, but is also influenced by the method of incorporation, non-deliquescent ingredients such as nitroaromatic compounds acting to some extent as waterproofing when the incorporation has been carried out hot. Deliquescence being a surface

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action also naturally depends on the size of the grain and on the porosity. In examining an explosive for deliquescence the most satisfactory method is to compare its behaviour in this respect with another explosive under similar circumstances. This is very simply done by spreading known weights on watch glasses and then exposing them to the atmosphere for the same number of hours and noting the increase in weight. As the humidity of the atmosphere varies from day to day it is better to expose the samples in a closed cupboard maintained at a constant temperature, the humidity of the air being maintained by flat dishes containing water or saturated solutions of salts.

Exudation

The British Home Office regulations for testing for exudation lay down that a section of the cartridge approximately equal in length to its diameter shall be cut so that the ends are flat and the edges sharp. This is then pinned on to a flat surface with its axis vertical by a pin passing through the centre and exposed to a temperature of 85° to 90° Fahr. (29·4° to 32·2° C.) without any wrapper for 144 consecutive hours, at the end of which time the upper cut surface must still be flat and the edges sharp, and the diminution of height must not exceed 25 per cent. American Straight Dynamites are very apt to exude nitroglycerine through absorption of water, and in the United States three tests are in use. In one the explosive is pressed for 1 minute between pads of cotton wool at 80 lbs. per square inch and must not show a loss of more than 3 per cent. In the second test it is submitted to centrifugal force when enclosed between cotton wool, and again in this case the loss must not exceed 3 per cent. Finally it is placed in a vertical position and heated for 6 days at 40° C.

International Committee

In 1912 the International Committee on Tests for Explosives recommended that the following tests should

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be applied to explosives in order to ascertain their safety for transport:—

I. *Preliminary Test*

Two samples each of ten grams of the non-desiccated explosive are placed in glass capsules fitted with a cover and having a diameter of 35 mm. and a height of 50 mm. The samples are heated to 75° C. for 4 hours, and at the end of that time no decomposition or change in appearance must have taken place, and no smell must be noticeable.

II. *Mechanical Shock*

The explosive is dried and powdered, and ·05–·10 gram is spread on a steel anvil in a layer 1·27 cm. in diameter and tested by the falling weight. The explosive must not be more sensitive than an equal weight of pure, dry powdered picric acid when tested under exactly similar conditions.

III. *Friction*

The explosive is ground in an unglazed porcelain mortar at a temperature of 25°–30° C., and its behaviour compared with that of picric acid under similar treatment.

IV. *Fire*

(*a*) Three grams of the explosive are tapped down into a glass test tube and its behaviour towards slow-burning safety fuse tested. If it ignites it must be classified as deflagrating and easily inflammable.

(*b*) The explosive is thrown into a hemispherical iron dish, 12 cm. in diameter, at a red heat. At first only ·5 gram of the explosive is used, but if this does not explode or ignite the amount is gradually increased to 5 grams.

(*c*) One hundred grams are placed on a sheet of asbestos board and then touched with an iron rod 15 mm. in diameter heated to a cherry-red heat (about 900° C.). The explosive should only burn slowly, and must not detonate. The ignition should die out when the hot rod is removed.

The Committee printed their recommendations in both French and English, but the English version is so

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ungrammatical that the foregoing description is abstracted from the French.

The Heat Test

The test known as the Heat Test was originally introduced by Abel for testing the stability of guncotton, and depends upon the time required to produce a given tint in starch-iodide paper when the explosive is heated under standard conditions. The exact conditions under which the test is carried out have been modified from time to time, but in 1909 the British Government appointed a departmental committee to investigate the whole matter and to specify standard conditions for the test as applied to explosives of various classes, and this committee presented its First Report in 1914. In Great Britain the Heat Test is the standard test of stability applied to all explosives, and unless an explosive satisfies this test it may neither be sold nor transported. Owing to the great importance lent to the test by these regulations the conditions under which it is carried out will be described in some detail.

In carrying out work with the heat test it must be borne in mind that it is exceedingly sensitive, and hence the most scrupulous cleanliness is absolutely essential. A special room must be devoted to it, the floor and walls of which are washed at frequent intervals, no reagents other than those required for the test should be allowed into the room, and the room should be situated as far as possible from the acid department. The room should be entered by as few people as possible, and the operator must wash his hands and rinse them with distilled water at frequent intervals. Direct sunlight should also be excluded from the room.

Most factories, especially those holding Government contracts, purchase their test papers from the Royal Gunpowder Factory at Waltham Abbey, but the following official regulations have been laid down for their preparation :—

The purest potassium iodide obtainable commercially is to be purified immediately before use by triple recrystallization from pure ethyl alcohol diluted with one-twentieth

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of its own volume of distilled water. The crystals are to be kept as small as possible and are to be dried in the dark on clean filter paper resting on a glass plate. When dry they are to be laid in a thin layer at the bottom of a platinum crucible and heated to dull redness for one minute, a spirit lamp burning pure alcohol being used.

The starch is to be best maize starch in the form of cornflour. Immediately before use it is washed six times with freshly distilled water, and then dried in a warm room in the dark on an unglazed porcelain plate.

The solution is prepared by suspending 3 grams of starch in 30 c.c. of distilled water and then pouring the suspension into 220 c.c. of freshly distilled boiling water contained in a Jena glass flask. The whole is then kept gently boiling, with occasional shaking, for 5 minutes, a spirit lamp burning pure alcohol being used. The solution thus obtained is poured into a solution of 1 gram of potassium iodide in 250 c.c. of distilled water, and the whole well mixed and allowed to stand overnight in a dark room. The following day the clear supernatant liquid is syphoned off and immediately used for dipping. This is done by pouring the solution into a porcelain tray reserved for the purpose, and then passing sheets of filter paper through it singly so that all except 3 cm. at the end of the strip passes beneath the surface of the liquid. The paper is then hung up by the dry part and a clean glass rod passed down each side to remove the excess of solution, after which it is allowed to dry in a warm dark room. When dry the undipped part is cut off, and also strips ·5 cm. wide round the edges, these portions being rejected. The remainder is then cut into rectangular slips 2 cm. long and 1 cm. wide and preserved for use in brown glass bottles. While cutting up the paper the operator is to wear clean cotton gloves, and the preparation of the paper is to be carried out in a special room reserved for the purpose and kept as dark as is compatible with the carrying out of the work. The Memorandum issued by the committee describes a method of testing the sensitiveness of the papers thus prepared.

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The quality of the filter paper used is of considerable importance, and the committee have as yet been unable to fix a definite specification, but suggest the following tentatively :—

(1) To consist entirely of pure normal cotton cellulose of strongly resistant quality and free from any loading or sizing.

(2) To have a smooth white surface, both sides being as nearly alike as possible.

(3) The average length of the fibres to be $2 \pm .5$ mm.

(4) The thickness to be $1.8 \pm .2$ mm. (Ciceri Smith's patent fixed pressure micrometer).

(5) The last treatment in its preparation to be a thorough washing with distilled water, and subsequent air drying in a pure atmosphere.

(6) To be free from all impurity, particularly acids, chlorine and peroxides.

(7) The loss of weight when boiled for 1 hour with 3 per cent. caustic soda solution not to exceed 7·5 per cent.

(8) It is not to produce more than 1·25 per cent. of its weight of cuprous oxide when heated to 100° C. for 15 minutes with Fehling's solution diluted with its own volume of boiling water.

(9) The sheets are to measure 50 cm. by 15 cm. and are to be packed in hermetically sealed tin cases holding 100 sheets each.

The standard tint is prepared by shaking ·48 gram of finest yellow ochre, ·2 gram raw umber and 5 grams of fine white gum arabic, all of which have been very finely ground in an agate mortar, with 100 c.c. of cold water until the gum is dissolved. After standing for 1 hour a stylographic pen is filled from the centre of the suspension, and with it lines not less than ·5 mm. or more than 1 mm. wide are ruled across filter paper.

This does not seem a very satisfactory method of producing a standard tint, as commercial pigments are apt to vary in shade, but a sealed pattern is kept at the Home Office with which comparisons can be made.

The apparatus for carrying out the heat test consists of a constant-level copper water-bath with seven equally

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spaced holes in the cover, one of these carrying the thermometer and the other six the test-tubes containing the explosives under examination. The level of the water is ¼ in. below the cover. The test-tubes are 5½ in. long, and have three etched marks on them at 3 in., 3⅜ in. and 5 in. from the bottom respectively. The lowest of these indicates the depth the tube is to be inserted in the bath, *i.e.* until this line is level with the cover, the middle one indicates the level of the lower moistened part of the test paper during the test and the top line the level of the lower side of the rubber cork. In carrying out the test the explosive is prepared as described below and transferred to the test-tube. A piece of test paper is then impaled on a platinum hook sealed on to a glass rod, this latter passing through a rubber cork, and the upper edge of the paper moistened with a mixture of equal volumes of distilled water and pure glycerine (B.P. specification). The rubber cork is then inserted into the test-tube, and the whole slipped into one of the holes in the cover of the water-bath and covered with an opaque cap to exclude light. The temperature of the bath is 160° Fahr. or 170° Fahr., depending on the explosive being tested, and the time is noted which elapses before the brown line which appears at the margin of the wet and dry portions of the paper matches the standard.

When setting up a test absolute cleanliness must be observed, and on no account must the paper be touched with the fingers, forceps being used.

In preparing explosives for the test, the end portions of cartridges or sticks are always rejected. The following outlines the methods employed for the different types of explosives:—

I. *Dynamite and other nitroglycerine preparations from which the nitroglycerine can be conveniently extracted with water.*

Thirteen grams of the explosive are evenly pressed down with a glass rod on a filter paper supported by a 5½-cm. glass funnel. The funnel is then placed in a heat test-tube and filled with water. This displaces the nitroglycerine of

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which 2 c.c. are collected, special heat test-tubes with a mark corresponding to this amount being used. If any water goes through with the nitroglycerine a fresh sample of the explosive must be extracted.

II. *Carbonite and similar friable preparations from which the nitroglycerine cannot be conveniently extracted with water.*

A sample weighing 3·2 grams is transferred to the heat test-tube and pressed down to a height of 3 cm. by means of a flat-headed glass rod.

III. *Cordite and other Propellants of Class* 3, *Division* 1. If the explosive is in sticks or tubes these are wiped with filter paper, the ends cut off and rejected, and the remainder then cut into pieces $\frac{1}{8}$ in. long and ground and sieved, a standard grinding mill and a standard nest of sieves being specified by the committee. The weight of the ground and sieved sample taken for the test is 1·6 grams, this not being pressed down but merely made to settle by tapping the outside of the tube. If the explosive is in grains it is not ground or sieved. Nitrocellulose propellants when in sticks or tubes are treated in the same way.

IV. *Nitrocellulose Pulp.*

The explosive is pressed between six thicknesses of filter paper, and 5 grams of the pressed pulp weighed out and dried at 48·9° C. on an aluminium tray for 15 minutes. The dried sample is then sieved under standard conditions, exposed to the air for 4 hours, and then 1·3 grams weighed out, transferred to the heat test-tube and pressed down gently to a height of 3 cm.

Nitrocellulose propellants in grains are tested in the same way as guncotton pulp, except that they are not sieved and the preliminary pressing between filter paper is of course dispensed with.

V. *Compressed Guncotton.*

Scrapings are taken from the centre of the slab by means of a horn spatulum and stirred for 15 minutes with cold distilled water. The water is then poured off and the process repeated, after which the guncotton is treated in exactly the same way as guncotton pulp.

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VI. *Bellite and Analogous Preparations.*

The weight taken is 1·3 grams, and this is gently tapped to the bottom of the test-tube.

VII. *Gelatinized Explosives.*

A sample weighing 3·2 grams is weighed out and ground up with 6·5 grams of French chalk in a Wedgwood pestle and mortar, the grinding being carried out at first with a squeezing and then with a circular movement of the pestle. With Blasting Gelatine the squeezing movement is carried on for 1½ minutes, with Gelatine Dynamite for 1 minute, and with Gelignite for ½ minute, the circular movement being in all cases maintained for ½ minute, after which the mass should be homogeneous in appearance. The mixture is gently pressed down in the heat test-tube to a height of 5 cm.

The committee found that different qualities of French chalk give different results when used for heat test purposes, but they were unable either to find a substitute or propose a definite specification. As a tentative measure they suggest the following :—

(1) It must not contain more than ·5 per cent. of moisture.

(2) Its bulk must be such that 50 c.c. without ramming or tapping weigh 23·5 ± 1 gram.

(3) It must pass a sieve composed of wire ·075 mm. in diameter and having 6400 meshes to the square centimetre, no rubbing being used.

(4) It must contain less than ·1 per cent. of soluble alkali calculated as $CaCO_3$.

(5) When warmed with hydrochloric acid it must give off at least ·25 per cent. of carbon dioxide, but not more than 1 per cent.

(6) It must not absorb more than ·5 per cent. of moisture when exposed for 24 hours to a saturated atmosphere at 15°–20° C. after being dried at 100° C.

Before use it is washed with distilled water, dried at 65°–70° C., and then exposed to a saturated atmosphere for 24 hours. This cannot be said to be a satisfactory definition, and it should not be impossible to obtain a more definite chemical compound suitable for the purpose.

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The following table shows the temperature and amount of the different explosives used, together with the minimum time in minutes they are required to stand :—

Explosive.	Quantity.	Temperature.	Time.
Nitroglycerine extracted from Dynamite	2 c.c.	160° F.	15
Friable nitroglycerine explosives ..	3·2 grams	160° F.	7
Gelatinized explosives	3·2 ,, +6·5 ,, French chalk	160° F.	10
Cordite and other nitroglycerine propellants	1·6 grams	160° F.	10
Nitrocellulose, pulp or compressed ..	1·3 ,,	170° F.	10
Nitrocellulose propellants	1·3 ,,	170° F.	10
Bellite, and the like	1·3 ,,	170° F.	10
Cordite Mark I. and Cordite M.D. (Government Contracts)	1·6 ,,	180° F.	30

The heat test has been adopted by most countries either alone or, more usually, in conjunction with other tests as the standard method of measuring stability, but the details of its application naturally differ in some respects from those in force in Great Britain. It would be a great convenience if an international method of carrying out the test could be established and adopted by all countries. The test suffers from the disadvantages of being too sensitive, and of being masked by minute traces of certain chemicals such as mercuric chloride. Alcohol and ethyl acetate prolong it, and consequently it is not very reliable for nitrocellulose propellants which have been gelatinized by these solvents. On the other hand, ozone or peroxide if present in minute traces greatly reduce it, and so does oxalic acid, although it probably does not affect the real stability.

As a manufacturers' guide the test is undoubtedly useful, but as a guarantee of stability far too much reliance would seem to be placed on it, as unless a very careful analysis of the explosive is made at the same time it is quite possible that really unstable explosives might pass the test, whereas really stable ones might fail. The test is really too sensitive, 135×10^{-6} mgr. of nitrogen peroxide being sufficient to produce the standard tint. It is difficult, however, to

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propose a more satisfactory test, although several attempts have been made to do so. These may be divided into three broad classes, viz. tests similar in nature to the Abel heat test, in which reliance is placed on the detection of traces of oxides of nitrogen; tests in which the explosive is submitted to more drastic treatment and which rely on the production of visible fumes, self-heating or explosion; and quantitative tests, in which the quantity of oxides of nitrogen is actually measured. Of the trace tests may be mentioned the Guttmann, Spica and Moir tests. Of these the first relies on the production of colour in a paper impregnated with a solution of diphenylamine and dilute sulphuric acid. It is not masked by mercuric chloride or by alcohol or ethyl acetate, and is in use in some countries, although its reliability has been criticized. Spica's test is similar, except that *m*-phenylenediamine and cane sugar are used, but it is far too sensitive. Moir's test, on the other hand, employs a mixture of α-naphthylamine, sulphanilic acid and acetic acid, and is even more sensitive.

Vieille's test involves more drastic treatment of the explosive, and a much less sensitive indicator is used, viz. litmus paper. It is carried out by heating the explosive on successive days for 8 hours to 110° C. or until the test paper assumes the standard tint. This process is repeated daily until the time is reduced to 1 hour, and the sum of all the times then taken as a measure of stability. Although officially adopted in France, the test must be regarded as a very crude one.

A somewhat more satisfactory test of a similar nature is used in Germany. In this test the explosive is heated under standard conditions at 135° C. (the boiling point of xylol), and the time noted which elapses before (1) litmus paper is reddened; (2) brown fumes become visible; (3) explosion takes place. If many samples are to be tested it is a somewhat tiresome test to carry out owing to the explosion.

Another test depending on the production of brown fumes has been introduced for testing Mark I. Service Cordite, which has been in stock for some time, and the heat test

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of which has fallen to between four and eight minutes at 160° F. The test is applied by placing 50 grams of the ground Cordite in a vacuum-jacketed silvered flask heated by a water-bath to 80° C. A sensitive thermometer is in contact with the Cordite, and the neck of the vessel is provided with a side tube. Observations are made of the interval of time that elapses before (1) brown fumes are seen in the side tube; (2) the Cordite begins to heat spontaneously; (3) the spontaneous rise in temperature reaches 2° C. The test is known as the Waltham Abbey Silvered Vessel Test.

Of the quantitative tests, the best known is that devised by Will. This is carried out by heating the explosive to 135° C. in a current of pure carbon dioxide, the rate of flow of the gas being 1000–1500 c.c. per hour. The carbon dioxide and gases evolved by the explosive are then led over heated cupric oxide and metallic copper in order to oxidize any organic matter and to reduce oxides of nitrogen to elementary nitrogen. Finally, they are collected over 40 per cent. caustic potash in order to absorb the carbon dioxide. The volume of nitrogen thus obtained is noted at regular intervals during the test, and from the figures thus obtained a very good idea of the rate of decomposition of the explosive can be formed.

Other tests of a similar nature have been devised, but they present little if any advantage over the Will test.

LITERATURE

Mechanical Shock

A friction test which has been adopted in America is described in the *Bulletin of the U.S Bureau of Mines*, No. 66, pp. 15 and 290.

The falling weight test is described in the following publications:—

"VI. International Congress of Applied Chemistry," vol. 2, p. 522.

"VII. International Congress of Applied Chemistry," Section III., p. 23.

S.S., 1906, pp. 14, 209. *Bulletin of U.S. Bureau of Mines*, No. 66, pp. 17, 287. Marine-Rundschau, 1905, 1345.

Detonation and Influence

Z. ang., 1904, 546.

P.S., 1913, p. 145.

"VII. International Congress of Applied Chemistry," vol. iii. *b*, p. 30.

Bulletin of the U.S. Bureau of Mines, No. 15.

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Exudation

U.S. Bureau of Explosives Reports, Nos. 2 and 4.
S.S., 1910, p. 213. *A.E.*, 1914, pp. 150, 162.

International Committee

"VIII. International Congress of Applied Chemistry," vol. 25, pp. 261, 305.

Heat Test, etc.

Very full directions for the application of the Heat Test will be found in "First Report of the Departmental Committee on the Heat Test as Applied to Explosives," published by Authority, London, 1914. This publication gives detailed drawings of the apparatus required. Some United States conditions will be found in A. Marshall, "Explosives," vol. ii., London, 1917, and in E. C. Worden, "Nitrocellulose Industry," vol. ii., London, 1911.

Several papers describing research work on the test have appeared from time to time, of which *J.S.C.I.*, 1910, 130, is of particular interest.

Guttmann described his test in *J.S.C.I.*, 1897, 293. See also *S.S.*, 1912, p. 153.

Moir's Test and Egerton's modification of it are treated in *J.S.C.I.*, 1913, 331, and 1914, 113.

Vieille's Test is described by R. Escales in "Schiessbaumwolle," p. 184, and in E. C. Worden, "Nitrocellulose Industry," vol. ii. p. 969.

The best descriptions of the German test at 135° C. will be found in R. Escales "Schiessbaumwolle," p. 183, and in E. C. Worden, "Nitrocellulose Industry," p. 971.

A. Marshall also gives a short description of it in his book "Explosives," vol. ii. p. 662.

The Waltham Abbey Silvered Vessel Test is described in "Regulations for Army Ordnance Services," published by Authority (1908).

Will's Quantitative Test has been studied by Robertson in *J.S.C.I.*, 1902, 819, and *Soc.*, 1907, 761. Further descriptions of the test will be found in—

R. Escales "Schiessbaumwolle," p. 186.
E. C. Worden, "Nitrocellulose Industry," p. 974.

The following references deal with other tests of a similar nature :—

Z. ang., 1904, 982 ; *J.S.C.I.*, 1905, 347 ; 1912, 161.

J. Am. Chem. Soc., **30**, 271 ; *Soc.*, 1907, 764. *S.S.*, 1905, p. 29 ; 1910, p. 121. *P.S.*, xiv. p. 42. *A.R.*, 1903, 26 ; 1904, 28 ; 1905, 28. "VIII. Congress of Applied Chemistry," vol. iii. *b*, pp. 147, 157. Also R. Escales, "Schiessbaumwolle," pp. 177–198; E. C. Worden, "Nitrocellulose Industry," pp. 968–984.

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CONCLUSION

In surveying the ingredients which so far have been found suitable for use in the manufacture of explosives, one is at once struck by the paucity of the number of oxidizing agents available. Of organic compounds only one, viz. nitroglycerine, contains more oxygen than is necessary for its complete oxidation. It is true that one or two compounds such as tetranitro methane contain an excess of oxygen, but they are difficult to obtain, and even if obtained have objectionable qualities such as intense toxic action. Nitromannitol, of course, has some available oxygen, but it is too expensive for general use, and other possible compounds such as dinitro tartaric acid are far too unstable. As it is theoretically impossible to obtain a nitroaromatic hydrocarbon with available oxygen, the unknown hexanitrobenzene would only contain just enough for its complete oxidation, the chances of obtaining a suitable organic oxidizing agent would seem very remote. Of the inorganic oxidizing agents available, only three classes are of any importance, viz. the nitrates, chlorates and perchlorates, although bichromates have been used to a very minor extent.

Of these the nitrates are the cheapest, but unfortunately the most suited of them, viz. ammonium nitrate, has the objectionable quality of being very hygroscopic. In spite of this it has met with wide application, and is probably destined to be employed to a far greater extent in the near future owing to the production of cheap ammonia by the Haber process and of cheap nitric acid by the oxidation of this or directly from the air by electrical means. Up to

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the present waterproofing the cartridges with wax has been chiefly relied on as a means of preventing the absorption of water by the explosive, although one or two explosives are put up in cases made of an alloy of tin and lead. These are objectionable, however, in underground workings owing to the poisonous nature of the lead oxide produced. Possibly aluminium foil may prove more suitable as its oxide is not poisonous, and the heat of combustion of the foil would considerably increase the power of the explosion. It should be noted that ammonium nitrate, with the exception of ammonium perchlorate, is the only inorganic oxidizing agent giving no solid products.

Of the other nitrates, sodium nitrate is the cheapest, but suffers from the same disadvantage as ammonium nitrate, only to a lesser extent. Potassium nitrate has not this disadvanatge, but is much higher in price. Neither barium nor lead nitrate is hygroscopic, but unfortunately both barium and lead salts are very poisonous, so that neither of these salts is suited for use as an ingredient in explosives for use underground, and in addition to its toxic properties, lead nitrate reduces the velocity of detonation to a great extent. As a class the nitrates, and particularly those of ammonium, sodium, and potassium, are likely to hold the premier place as oxidizing constituents in explosives. In this connection it should be pointed out that the synthetic nitric acid processes are at present more suited to the production of nitrates than to the production of nitric acid. In all these processes it is not nitric acid but nitric dioxide that is produced, and although this can be converted into nitric acid by the action of water and air—

$$3NO_2 + H_2O = 2HNO_3 + NO$$
$$NO + O = NO_2$$

this can only be done at present by means of an elaborate system of towers, the gases flowing in one direction and the liquor being circulated from the bottom of one tower to the top of the next on the counter-current principle. Pumping straight nitric acid is always troublesome, and although

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it can be achieved with ferro-silicon pumps or in glass or stoneware pipes by the air-lift, the former are expensive and the latter inefficient. In any case, the towers yield an acid of a strength not exceeding 60 per cent., which is too weak for most nitrations, and it can only be concentrated by distillation with sulphuric acid, calcium nitrate or other suitable dehydrating agent. The methods of concentration will no doubt be greatly improved in the near future, but in the meantime the weak acid is quite suitable for making nitrates, as it is only necessary to neutralize it with the base and then evaporate the solution. Of course only a small proportion of the nitrate made will be used for the production of explosives, the bulk being manufactured for agricultural purposes. It may be remarked in passing that the use of synthetic nitric acid will go a long way towards simplifying the chemical engineering side of plant construction, as the synthetic acid, unlike that obtained from Chili saltpetre, contains no chlorine and is consequently much less corrosive. Probably chrome steel will be found quite suitable for handling it, although this alloy is badly attacked by commercial nitric acid made from Chili saltpetre.

Of the chlorates, only the potassium salt is used to any extent, the sodium salt being too deliquescent and the ammonium salt unstable. Even the potassium salt is never likely to be much used except for the production of matches, as Cheddite is about the only chlorate mixture which is not unduly sensitive, or likely to become so on keeping.

The perchlorates are more stable than the chlorates, from which they are obtained by electrolysis, and in addition they contain more oxygen. Before the war their use was on the increase, and the erection of a large factory for their production for war purposes should lead to their increased application. It is interesting to compare the prices of nitrates, chlorates and perchlorates, and the following figures for the potassium salts are based on prices current in 1910. The figures are in shillings per 100 lbs. of salt and in shillings per lb. of available oxygen :—

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	Price per cwt. Salt.	Per cent. Available Oxygen.	Price per lb. Available Oxygen.
KNO_3 ..	.. 47	40	1·17
$KClO_3$ ..	.. 66	39·3	1·68
$KClO_4$ ..	.. 80	46·3	1·73

The fact that the perchlorate is slightly more expensive than the chlorate per unit available oxygen is more than counterbalanced by increased safety, but it is difficult to see how perchlorates can ever compete with nitrates for general purposes, especially in view of the production of cheap nitric acid in the near future, although they will no doubt be increasingly used for special purposes. Ammonium perchlorate, like ammonium nitrate, gives no solid matter, but unfortunately it gives chlorine, and so can only be used in conjunction with an alkali or alkali earth nitrate or organic salt to provide a base with which the chlorine can combine. It is practically non-hygroscopic, and cost (pre-war) about 5¾*d.* per lb., this corresponding to 3·8 shillings per lb. of available oxygen. This figure must not be compared with those given above for the potassium salts as ammonium perchlorate is an explosive in itself—

$$2NH_4ClO_4 = 4H_2O + Cl_2 + N_2 + 2O_2$$

In spite of the disadvantage of producing chlorine the salt is likely to find considerable application, as it seems to assist the detonation to spread provided a strong enough detonator or primer is used.

Turning now to the combustible matters, these are chiefly nitroaromatic hydrocarbons, and to produce a really satisfactory explosive they should for preference be capable of exploding alone. At present the nitro derivatives of benzole and toluol are chiefly used, and to a lesser extent the nitro derivatives of naphthalene. This latter hydrocarbon, like all condensed nuclei compounds, suffers from the disadvantage that the number of nitro groups that can be introduced is smaller in proportion to the number of carbon atoms than is the case with uncondensed nuclei, and for this reason derivatives of fluorene, acenaphthene, pyrene, etc.,

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although they will probably come into use in the dye stuff industry, are not likely to be employed in the manufacture of explosives even if produced at a sufficiently low price. This is made clear in the following table, in which is shown the number of atoms of oxygen per molecule of compound and the grams of oxygen per hundred grams of compound which are necessary for complete oxidation :—

Compound.	Molecular weight.	Extra Oxygen required.	
		Atoms per mol.	Grams per 100 grms.
Dinitrobenzole	168	10	94·4
Trinitrobenzole	213	7½	56·4
Dinitrotoluol	182	13	114
Trinitrotoluol	227	10½	74
Trinitronaphthalene	263	16½	100
Tetranitronaphthalene ..	308	16	83·1

Trinitrobenzole shows up best, but unfortunately it is too expensive to produce. It is notable that no catalyst for facilitating nitration has yet been discovered. Such a catalyst would be most valuable, and might have a great influence on explosives manufacture.

It seems improbable that for general purposes much progress is to be expected in the way of production of new combustible matters, although exhaustive nitration may, and probably will, lead to the production of highly nitrated compounds like tetryl which, although suitable for special purposes such as for initiators, are too expensive for general blasting use. The numerous trinitrotoluol plants put down for war purposes and the experience gained in the manufacture of this substance render it probable that it will become the standard combustible. In any case, the amount held in stock for military purposes will probably suffice to supply the needs of the explosives works for some time if it is disposed of as " surplus stores."

Up to the present Gelignite has been the standard high explosive for general blasting purposes, and nitroglycerine explosives have also been very widely used in coal mines, but it is doubtful if nitroglycerine explosives will retain their

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supremacy much longer. The production of cheap ammonium nitrate and cheap T.N.T. will certainly give a great impetus to explosive mixtures composed of these ingredients, and as their manufacture is cheap and safe so that very little explosion risk has to be carried it should be possible to put them on the market at a very low price, thus enabling their use to become more general, *e.g.* for agricultural work. Whether the Merger Company (Explosives Traders, Ltd.) which now controls practically the whole explosives trade in Great Britain will do so or not remains to be seen. If not it is probable that the big mine owners will manufacture their own explosives, more especially as the big coal mine companies usually also own by-product coke oven installations, and will no doubt be among the first to instal synthetic ammonia plants. Mixtures of T.N.T. and ammonium nitrate can be made up to be as powerful as Gelignite, and by adding aluminium powder extremely powerful explosives of the Ammonal class can be obtained. Of course there is always the difficulty of deliquescence, but the present waxed paper wrappers are more or less satisfactory and are capable of improvement, and the introduction of aluminium foil cases would seem to be quite feasible.

The future is likely to see considerable improvement in detonators, and probably the ordinary fulminate-chlorate detonator will be very largely replaced by the composite detonator made with lead azide, unless further research brings to light a more satisfactory initiator.

The probable development of the propellant branch of the industry is more difficult to forecast, although minor improvements will be brought about in manufacturing details. No great departure from present day composition is to be looked for until some smokeless oxidized agent is discovered which has suitable properties. Of the only two available at present, ammonium nitrate is far too deliquescent, and ammonium perchlorate corrodes the barrel too much to be used. Unless such an oxidizing agent is found any great departure from present day compositions could only be made by the introduction of some " combustible matter " containing sufficient

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oxygen to explode without the production of smoke, *i.e.* containing enough oxygen to convert the hydrogen into water and the carbon into monoxide. Both trinitrobenzene and tetryl almost satisfy these conditions, and an almost smokeless powder could be produced from them by adding a little potassium nitrate. Unfortunately such mixtures are far too brisant for use as propellants, and at present it is not easy to see how their combustion is to be regulated. A propellant made from definite pure chemical compounds would have many advantages over those composed of indeterminate mixtures like nitrocellulose, but no success has yet been achieved in this direction. Of course, nitro starch may become a commercial success, but it is little more definite in nature than nitrocellulose.

In recent years a considerable number of patents have been taken out covering the use of stabilizers, and more progress is to be looked for in this direction, a stabilizer being desired which does not have a bad effect on the ballistic properties of the propellant and which does not mask stability tests, thus giving rise to a false belief in security.

The war has shown to what an extent the dyestuffs, explosives and fertilizer trades are interdependent, and the advent of synthetic nitrates will make them even more so. It is notable that one of the largest explosive producers in the world, E. A. du Pont de Nemours (U.S.A.), is now concentrating on synthetic dyestuffs, and that in this country Nobel's Explosives Company, Ltd., now merged into Explosives Trades, Ltd., have acquired a large interest in the dyestuff combine. Whether this tendency to concentrate business in the hands of one combine will ultimately benefit the consumer or not is doubtful, although it will no doubt benefit the shareholders.

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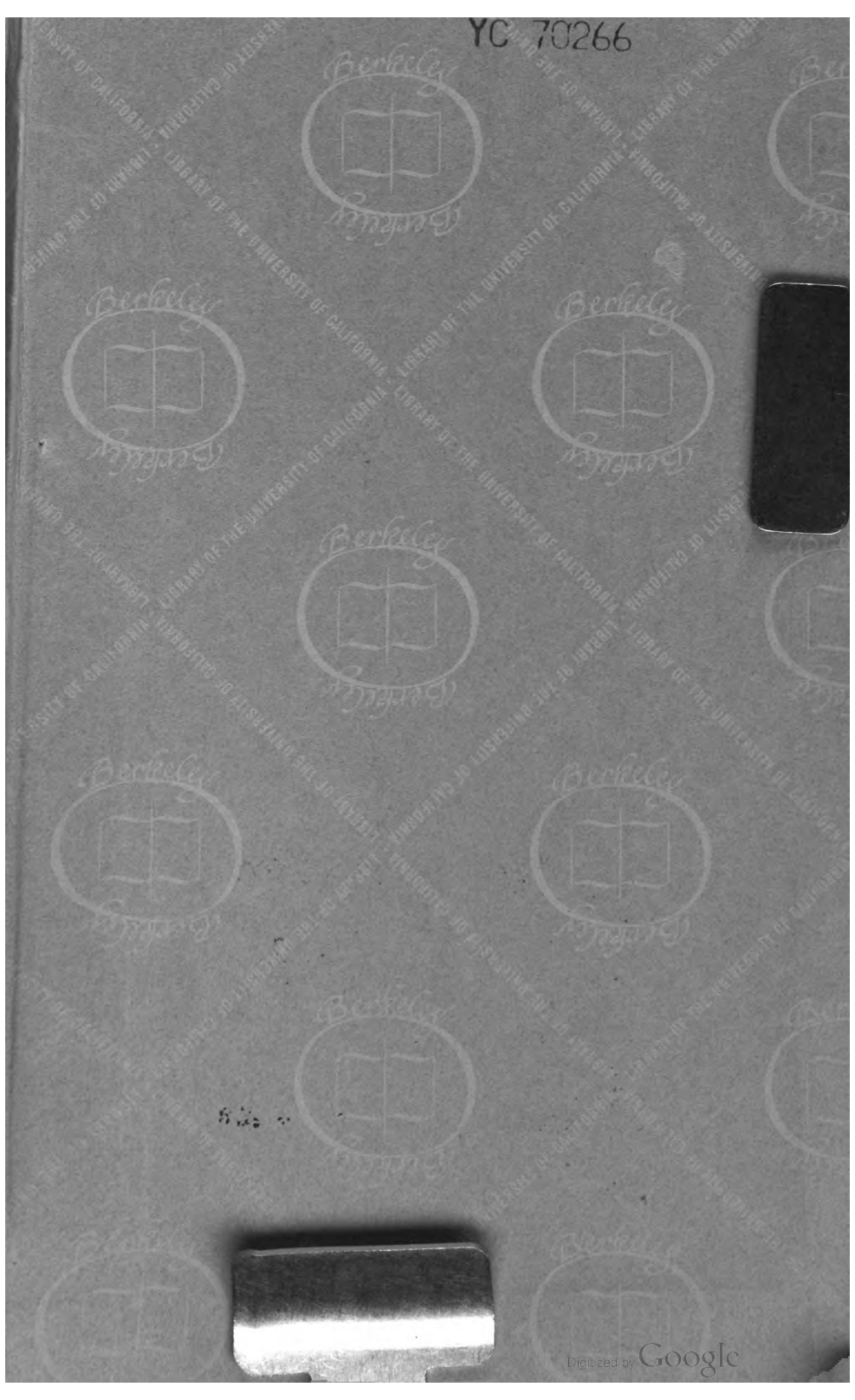

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